Final Sampling and Analysis Plan for the Remedial Investigation

Gateway National Recreation Area
Queens, New York

Spring Creek Park Site
EDL #5NER3348

December 4, 2024

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Gateway National Recreation Area, New York
National Park Service

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Signatories:

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By signing above, I attest that I meet the qualifications of an Environmental Professional as defined under 40 CFR §312.10(b) and that this document was prepared by me or under my supervision/responsible charge.		

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Table of Contents

List of Figures

List of Tables

List of Appendices

List of Abbreviations and Acronyms

µCi	microcurie
µg/L	microgram per liter
µR/hr	micro-Roentgens per hour
bgs	below ground surface
bss	below the sediment surface
°C	Celsius
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CH_4	methane
CO	carbon monoxide
CO_2	carbon dioxide
COC	chain of custody
COPC	contaminant of potential concern
COPEC	contaminant of potential ecological concern
cpm	counts per minute
CSM	conceptual site model
CSRM	coastal storm risk management
DGL	downhole gamma logging
DoD	Department of Defense
DPT	direct push technology
DQI	data quality indicator
DQO	data quality objective
DU	decision unit
EB	equipment blank
EDD	electronic data deliverable
EDL	Environmental and Disposal Liabilities
EE/CA	Engineering Evaluation and Cost Assessment
ELAP	Environmental Laboratory Accreditation Program
EPC	exposure point concentration
ESV	ecological screening value
FEMA	Federal Emergency Management Agency

FID	flame ionization detector
FR	Federal Register
FSP	Field Sampling Plan
ft	feet or foot
G2S	a joint venture between Tidewater, Inc. and SDC
Gateway	Gateway National Recreation Area
GIS	geographic information system
GKP	Great Kills Park
GMP/EIS	General Management Plan/Environmental Impact Statement
GOB	Gravesend and Oldmills
GPS	global positioning system
GWS	gamma walkover survey
H_2S	hydrogen sulfide
HAZWOPER	Hazardous Waste Operations and Emergency Response
HHRA	human health risk assessment
HMGP	Hazard Mitigation Grant Program
HRE	Hudson-Raritan Estuary
HQ	hazard quotient
HTRW	hazardous, toxic, and radiological waste
I.D.	inner diameter
ID	identification
IDQTF	Intergovernmental Data Quality Task Force
IDW	investigation derived waste
JBEC	Jamaica Bay Environmental Conference
JCO	The Johnson Company
LDPE	low density polyethylene
LNAPL	light non-aqueous phase liquid
LOD	limit of detection
LOQ	limit of quantitation
MCL	Maximum Contaminant Level
mg/kg	milligram per kilogram
mm	millimeter
mR/hr	milliroentgens per hour
mrem	millirem

MS/MSD	matrix spike/matrix spike duplicate
NAB	USACE Baltimore District
NaI	sodium iodide
NAN	USACE New York District
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NE	not established
NEPA	National Environmental Policy Act
NFG	National Functional Guidelines
NJDEP	New Jersey Department of Environmental Protection
NIST	National Institute of Standards and Technology
NNBFs	natural/nature base features
NOAA	National Oceanic and Atmospheric Administration
NPS	National Park Service
NUREG	USNRC technical report designation
NWI	National Wetlands Inventory
NYCDEP	New York City Department of Environmental Protection
NYCDP	New York City Department of Parks
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
NYT	New York Times
O.D.	outer diameter
O_2	oxygen
ORP	oxidation-reduction potential
PAH	polycyclic aromatic hydrocarbon
PAL	project action levels
PCB	polychlorinated biphenyl
pCi/g	picocuries per gram
pCi/L	picocuries per liter
PID	photoionization detector
PM	project manager
PPE	personal protective equipment
ppm	parts per million
PRGs	Preliminary Remediation Goals
PVC	polyvinyl chloride

QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
QSM	Quality Systems Manual
RI	Remedial Investigation
RPP	Radiation Protection Plan
RSE	Removal Site Evaluation
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SCP	Spring Creek Park
SDC	Sustainable Design Consortium
SDG	sample data group
SG	soil gas
Site	Spring Creek Park Site
SLERA	screening level ecological risk assessment
SOP	standard operating procedure
SPLP	synthetic precipitation leaching procedure
SU	sampling unit
SVOC	semi-volatile organic compound
TB	trip blank
TOC	total organic carbon
TPH	total petroleum hydrocarbons
TSCA	Toxic Substance Control Act
U.S.C.	United States Code
UCL	upper confidence limit
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USACE	United States Army Corps of Engineers
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
USGS	United States Geologic Survey
USNRC	United States Nuclear Regulatory Commission
VISL	vapor intrusion screening level
VOC	volatile organic compound
VSP	Visual Sampling Plan

1 Introduction

This Sampling and Analysis Plan (SAP) is for Remedial Investigation (RI) activities for Spring Creek Park (SCP) located in Queens, New York. SCP is an approximately 237-acre area managed by the National Park Service (NPS) as a part of the Jamaica Bay Unit of the Gateway National Recreation Area (Gateway) (**Figure 1-1**). This SAP was prepared on behalf of NPS by G2S (a joint venture between Tidewater, Inc. and the Sustainable Design Consortium [SDC]), under contract with the United States Army Corps of Engineers (USACE), per Contract No. W912DR-20-D-0022, Delivery Order W912DR21F0048. RI sampling activities are being undertaken at SCP pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 United States Code (U.S.C.) §§ 9601 *et seq.*, and its associated regulations, the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 Code of Federal Regulations (CFR) Part 300.

The Site consists of the entire 237-acre area of SCP. Waste disposal activities were performed at the Site from 1948 to the early 1960s. From the late-1950s to the late-1960s/mid-1970s, sewage sludge was mixed with sand and clay to create a soil amendment (artificial topsoil) to cover the ground surface after the completion of waste disposal activities.

In the mid-1990s, SCP was identified for potential ecological restoration to improve the Jamaica Bay ecosystem. Site soil was investigated in 2001 as part of a feasibility study for the ecological restoration (USACE, 2013). The original restoration plans were later adapted as part of a Federal Emergency Management Agency (FEMA) Hazard Mitigation Grant Program (HMGP) award to include natural/nature base features (NNBFs) to provide coastal storm risk management (CSRM) benefits and enhanced coastal resiliency to the adjacent Howard Beach Community (USACE, 2017a). Several investigations were carried out to advance the FEMA HMGP project, including (1) a 2016 Phase I Archeological Sensitivity Assessment that included geotechnical soil borings (HDR, 2016) and (2) a 2017 Site Investigation (SI) and Screening Level Ecological Risk Assessment (SLERA) which evaluated surface and subsurface soil based on proposed excavation cut elevations to determine ecological risk from soil that would be newly exposed and options for managing the proposed excavation materials (USACE, 2017a). In 2018, the proposed project underwent detailed design and an Environmental Assessment under the National Environmental Policy Act (NEPA) (Princeton Hydro, 2019).

A limited gamma radiation walkover survey of NPS-maintained park fire roads (referred to as the fire road) that allow for pedestrian access was conducted in 2017 (USACE, 2018). This survey was performed due to historical similarities between SCP and Great Kills Park (GKP), where radiological contamination was determined to be associated with waste disposal operations analogous to those conducted at SCP (NPS, 2019). The survey identified elevated levels of radiation, including five radiological artifacts (deck markers) that were recovered and removed from the Site (USACE, 2018).

In 2017, NPS conducted a Removal Site Evaluation (RSE) that determined the release of hazardous substances at the Site potentially posed a threat to public health, welfare, and the environment (NPS, 2017). Based on these findings, NPS conducted an Engineering Evaluation and Cost Analysis (EE/CA) to support the implementation of a removal action under CERCLA. The EE/CA was carried out between 2018 and 2020 and involved investigations of groundwater, subsurface soil, surface water, and sediment (The Johnson Company [JCO], 2020). The EE/CA investigation was designed to a determine if

contamination in Site groundwater and surface water would pose a potential unacceptable human and/or ecological risk after the implementation of the HMGP project. A key assumption of the EE/CA was that soil cover proposed for the Site under the HMGP project would prevent contact with contaminants present in waste fill; therefore, surface soil was not investigated. The EE/CA also documented two limited gamma walkover surveys completed in 2018 and 2019. These surveys covered (1) a seven-acre area that burned during an April 2019 brush fire as well as (2) a four-acre area where donated sand is now stockpiled. One man-made radiological artifact was located and removed from the stockpile area. In 2019, FEMA decided not to fund the HMGP Project.

In response to FEMA's decision not to fund the HMGP Project and due to the uncertainty of future Site conditions, NPS's approach under CERCLA is to conduct an RI to evaluate the nature and extent of Site-related contamination in soil, groundwater, sediment and soil gas, evaluate the nature of radiological anomalies, evaluate the presence of landfill gas, and evaluate concentrations of contaminants in the identified reference area. This RI SAP defines:

- The purpose of this study;
- The use for the data generated;
- The quality of data needed to accomplish the goals of this study; and
- The data collection methods.

Once the Site has been adequately characterized, the NPS's approach under CERCLA is to proceed with an RI Report.

1.1 CERCLA and National Park Service Authority

This SAP was generated in accordance with the NPS SAP template (NPS, 2018a), United States Environmental Protection Agency's (USEPA) *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006), *Guidance for Quality Assurance Project Plans* (USEPA, 2002), *EPA Requirements for Quality Assurance Project Plans* (USEPA, 2001a), and the *Intergovernmental Data Quality Task Force's (IDQTF) Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP)* (IDQTF, 2012). NPS is authorized under CERCLA, 42 U.S.C. §§ 9601 *et seq.*, to respond as the Lead Agency to a release or threatened release of hazardous substances and/or a release or threatened release of any pollutant or contaminant that may present an imminent and substantial danger to public health or welfare on or from land under the jurisdiction, custody, or control of NPS.

CERCLA's implementing regulations, codified in the NCP, 40 CFR Part 300, establish the framework for responding to releases and threatened releases of hazardous substances. The NCP prescribes two processes for responding to releases: removal actions and remedial actions (See NCP Sections 300.400 through 300.440). Based upon the results of preliminary investigations performed by NPS in 2019, NPS has determined that conducting a remedial action, specifically a Remedial Investigation (RI) and Feasibility Study (FS), was the appropriate next step in the CERCLA process for the Site.

A SAP is required because environmental samples will be collected during the completion of the RI (See NCP Sections 300.415 and 300.430). This NPS SAP is comprised of multiple sections which include the Field Sampling Plan (FSP - **Section 5**) and the Quality Assurance Project Plan (QAPP - **Sections 2, 3, 4,**

6, and 7). The FSP describes the number, types, and locations of samples as well as the types of analyses that will be conducted on the samples. The QAPP describes the project's policy, organization, and functional activities as well as the data quality objectives (DQOs) and measures necessary to achieve the goals of the study. A standalone QAPP is not necessary because quality assurance/quality control (QA/QC) measures are identified for each specified procedure.

In addition, NPS has a number of regulations that apply to the release of hazardous substances on NPS land (see NPS, 2014a), including the NPS Organic Act of 1916 (54 U.S.C. §100101 *et seq*. 36 CFR Part 1), which requires that NPS manage parks to conserve the scenery, natural and historical objects, and wildlife and to provide for their enjoyment by such means as will leave them unimpaired for the enjoyment of future generations. Therefore, whether the Site poses risks to the interaction of organisms and the environment is especially relevant to the NPS responsibility to protect park resources.

Because radioactive materials have been encountered at the Site, the United States Nuclear Regulatory Commission (NRC) is an important stakeholder at the Site. Therefore, a Memorandum of Understanding (MOU) between NPS and NRC was established August 17, 2020. The purpose of the MOU is to describe the intent and plan of the two agencies to work together to address their overlapping statutory authorities for addressing radioactive material during the response actions at the Site. Provisions of the MOU with NRC lay out each agency's roles and responsibilities, including those associated with communications, planning, access to the Site, records, and more.

1.2 Purpose of Field Sampling

The purpose of this field sampling event is to generate representative data of adequate quality to evaluate the nature and extent of Site-related contamination within soil, groundwater, sediment and soil gas; evaluate the nature of radiological anomalies identified at the Site; evaluate the presence of landfill gas; evaluate concentrations of contaminants in the identified reference area that are potentially attributable to background sources. Specific DQOs for the field sampling are presented in **Section 4** of this SAP.

This SAP describes the following field investigation activities: surface and subsurface soil sampling (Site and reference area); installation and development of groundwater monitoring wells (historical support areas at the Site); sediment sampling (Site); installation and sampling of soil gas probes (Site); Gamma walkover surveys (Site); and focused investigations of radiological anomalies in waste fill (Site).

1.3 Site Location

SCP occupies the area between Jamaica Bay and the Howard Beach Community in the Borough of Queens, New York City, New York (see **Figure 1-1**). The Site is bounded by the Belt Parkway to the north, the Howard Beach Community (78th Street, 161st Avenue, 83rd Street, and165th Avenue) and Cross Bay Boulevard to the East and Jamaica Bay to the south and west. Coordinates for the approximate center of the Site are 40° 39' 05.4" N, 73° 50' 56.8" W. Access gates are located at 159th Avenue, 164th Avenue, and 92nd Street. The approximate extent of the Site is shown in **Figure 1-2**. The Site Environmental and Disposal Liabilities (EDL) number is 5NER3348.

2 Site Description, Previous Investigations, and Conceptual Site Model

This section summarizes the known environmental information and historical activities that have occurred at the Site and presents this information in the form of a graphical Conceptual Site Model (CSM). The development of a clear and thorough CSM is a critical component for ensuring that key Site elements are considered before any samples are collected, gaining stakeholder approval, and assisting the Planning Team in developing the DQOs (**Section 4**), as well as assisting the field team in making decisions in the field.

2.1 Key Site Features

This section includes a summary of information related to the Site, including the operational history, waste characteristics, geology and hydrogeology, hydrology, local climate, and sensitive environments.

2.1.1 Site Description

SCP is located in Queens, New York on waterfront property between Cross Bay Boulevard and the Belt Parkway. SCP is within the Jamaica Bay Unit of Gateway. SCP is approximately 237 acres in size and is directly adjacent to Jamaica Bay and Old Mill Creek (**Figure 1-2**). The Jamaica Bay Unit is one of three units that comprise Gateway—the Jamaica Bay Unit in Brooklyn and Queens Counties, the Staten Island Unit, and the Sandy Hook Unit (northern shore of New Jersey) (**Figure 1-1**).

Prior to waste disposal activities, the area of SCP consisted of open water, marshland, and marshland that had been historically altered through the placement of hydraulic fill. The Site shoreline has a unique indention point referred to as “the Cove” which marks the outlet of the historical Crum Hill Creek. The Cove serves as a natural break point to define a southern portion and northern portion of the Site. The shoreline shape of the southern portion of the Site is entirely man-made and was created by large sand dikes used to prepare the Site for waste disposal activities. Much of the southern shoreline of the Site is sandy dunes. The shoreline of the northern portion of the Site appears not to have been prepared in the same manner and is primarily a marsh shoreline.

During previous investigations at SCP, chemical and radiological contamination was identified at the Site. The Site is primarily composed of waste fill derived from New York City Department of Sanitation’s historical waste disposal operations at the Site, hydraulic fill, and soil amendment (containing sewage sludge). During historical operations, hydraulic fill was used to cover the waste materials and reduce odors. Hydraulic fill was also used to construct the containment sand dikes as well as to fill submerged areas to above the high-water line prior to placement of waste. A soil amendment (artificial topsoil) was made from sewage sludge and clay and was applied across the Site to cover the fill and to facilitate vegetative growth.

Environmental investigations in 2002, 2017, and 2020 identified chemical and radionuclide contamination in multiple environmental media. Near-surface radiological artifacts were identified and removed during environmental investigations in 2017 and 2018.

The Site is being investigated as one 237-acre area. Within the Site, there are two areas identified as historical support areas where vehicles were staged, or buildings existed to support the main historical operations at the Site. Historical support areas include the area south of 92nd St shown in the 1951 aerial imagery (1951 support area) and the area west of 165th Avenue shown in the 1954 aerial imagery (1954 support area). In addition, potential future land use plans for SCP designate an area for recreation along the boundary of Site adjacent to the residential area, with the remainder of SCP designated as a natural area (**Section 2.3**).

2.1.2 Operational History

The operational history of SCP is presented in three epochs defined as: prior to, during, and after the primary waste disposal operation which took place at the Site from 1948 through the mid-to-late 1960s.

Prior to Waste Disposal Operations (Pre-1948)

Early topographic maps from around 1900 identify the area later to become SCP as consisting of open water and marshes (**Figure 2-1**). An atlas from 1907 supports this original state and indicates the earliest and only settlements within the Site were in the northwesternmost portion (**Figure 2-1**). There are no other records of historical residential use of the Site. The construction of the Rockaway Railroad Trestle in the 1880s and the extension of the Long Island Railroad to the Rockaways around this same time encouraged development in the surrounding areas (HDR, 2016).

In the early 1900's, city planners envisioned Jamaica Bay becoming an ocean port and commercial and industrial center. The scheme was originally referred to as the Jamaica Bay Improvement Plan which called for the elimination of all marshes and meadows within the Bay and the creation of two large entirely bulkheaded islands. A ship channel was to be a circular canal going around the two islands. Periphery lowlands were to be filled in and bulkheaded. (Jamaica Bay Environmental Conference [JBEC], 1984). There would be 15 basins and 30 or 40 piers for vessels. **Figure 2-2** provides illustrations of the early plan. A later-adjusted version of the port plans is shown in a wayside exhibit highlighting the "Great Port Scheme" (**Figure 2-2**) (NPS, 1990). Plans for the great port failed with Canarsie Pier being the first and only portion realized. Plans to develop Jamaica Bay into a port were ultimately abandoned in favor of plans proposed and promoted by Robert Moses to transform the cities shore areas into parks and parkways (NPS, 1990). However, the legacy of the port scheme was the normalization of the large-scale land modifications around Jamaica Bay of which included SCP.

Land changes in the areas nearby the Site occurred incrementally from the 1900s to the early 1920s with dredging of ship basins, dredging of shipping channels within Jamaica Bay, and filling marshes with the dredged material. For example, just east of the Site, Shellbank Basin was created in the mid-1920s as documented by 1924 aerial imagery (**Figure 2-3**). In the 1930s, dredging and land modification around Jamaica Bay, as well as other coastal shorelines of the city increased dramatically under the direction of Robert Moses. It was during this period that the remaining shore areas owned by the City were slated for recreation and residential development as well as the construction of Shore Parkway (Belt Parkway) (HDR, 2016) which serves as the northern boundary of the Site.

Early aerial photographs from 1924 also show that a roadway referred to as Flynn's causeway was under construction running through the Site at that time (**Figure 2-3**). Flynn's causeway was constructed by

Patrick Flynn, a local entrepreneur, with the intent of connecting the waterfront with settlements to the north. Although Flynn's causeway was never fully realized, fragments of its remains are still visible at low tide at the southeastern sector of SCP (HDR, 2016). This construction resulted in historical filling of portions of the Site near the outfall of what was referred to in early atlases as Crum Hill Creek located at the main indention point along the western shoreline of the Site, also referred to as "the Cove". The section of Flynn's causeway located in the southern portion of the Site also filled in areas of marsh that extended into open water to the South. In 1924, construction of Cross Bay Boulevard, which serves as the Eastern boundary of the Site was under way and filling to support the Cross Bay bridge abutment is visible in the 1924 aerial imagery (**Figure 2-3**). A topographic map from 1947, just prior to waste disposal operations, shows the new bridge abutment and substantial new marshland created immediately west of the abutment (**Figure 2-3**). The 1947 topographic map shows the state of the Site close in time to the beginning of the waste disposal operations.

These specific construction and filling activities provide information on areas of the Site which were altered prior to waste disposal operations and suggest there may be less or more waste fill present in certain areas of the Site. Around 1948, prior to the start of waste disposal operations, the present-day Howard Beach neighborhoods of Rockwood Park and Spring Park did not exist and residential settlement was in the early stages of spreading west of Cross Bay Boulevard. Geospatial analysis of the 1924 aerial imagery and the 1947 topographic map indicates that prior to the start of waste disposal operations in 1948, the 237 acres within the current extent of SCP were comprised of approximately 109 acres (46%) of open water, 81 acres (34%) of marsh, and 47 acres (20%) of historically filled areas.

Waste Disposal Operations (1948-1960's)

The plans for and implementation of waste disposal operations at SCP are tied to the larger history of garbage disposal practices for the City of New York. A Supreme Court decision banned New York City from ocean garbage dumping in 1934 citing it created a public nuisance, forcing the City to find a new solution for trash disposal (New York Time [NYT], 1955). The City started a program of "land reclamation with garbage and refuse being used for fill until incinerators could be constructed" (NYT, 1955).

It was through the program of land reclamation, spurred by the Supreme Court decision, that New York City Department of Parks (NYCDP), Parks Commissioner, Robert Moses, began implementing his vision of creating public parks from "waste lands and swamps" (NYCDP, 1950). In a 1950 report to the Mayor, the NYCDP reports that, "We have used every kind of filling material – hydraulically dredged sand, ship ballast, rubble from bombed buildings in England and from the demolition of structures within the city, Department of Sanitation garbage, refuse and ashes, rock from deep cellar excavations and excess stuff from public, semi-public and private residential developments. (NYCDP, 1950)"

Prior to the beginning of waste disposal operations at SCP, there were several dumps already operating in Queens, including College Point, Rosedale, and Edgemere dumps (Long Island Star-Journal, 1948d). However, citizens complained about the dumps as being a nuisance and having bad odors (Long Island Star-Journal, 1948b). An idea was proposed to develop a modern "Super Dump" in Queens at SCP that would have enough capacity to replace the existing dumps in Queens until incinerators could be built (Long Island Star-Journal, 1948a). The "Super Dump" was to be odorless and employ the latest technical innovations to prepare the Site and manage the operations (Long Island Star-Journal, 1948b). Unlike other

dumps in Queens, the Spring Creek "Super Dump" was run by the NYCDP. Historically, similar operations had been run by the New York City Department of Sanitation and were in large part badly controlled and offensive (NYCDP, 1950).

At the Spring Creek "Super Dump", Department of Sanitation collection trucks dumped their loads and the NYCDP staff would bulldoze the trash to systematically fill lands, while a three-person crew of exterminators managed pests, such as rats (Long Island Star Journal, 1962). Odors were controlled by sand being immediately placed on top of garbage (Long Island Star Journal, 1962). Sand to cover trash was sourced from Jamaica Bay and stored in long stockpiles on Site (Long Island Star Journal, 1948b, 1962; Wave, 1951). In addition to covering the trash with sand to reduce odors, the NYCDP also sprayed disinfectant and deodorant daily (Long Island Star Journal, 1962). The chemical makeup of the disinfectants and deodorants is unknown. Orthodichloro-benzene was historically used at GKP as a disinfectant (NPS, 2018b); however, it's use at SCP has not been confirmed. Proposals for the original 125-acre waste disposal area at SCP specified a 3-year time frame to complete the filling and included plans to convert the area to a public park once waste disposal operations were complete (Long Island Star-Journal, 1948a).

The NYC Board of Estimates approved the plan to develop SCP for waste disposal operations in August 1948. The operation would be situated on city-owned waterfront property between Cross Bay Boulevard and the Brooklyn borough line. The resulting reclaimed land would become an addition to an existing park, already known as Spring Creek Park just over the Brooklyn line (Long Island Star-Journal, 1948d). By September, bids were solicited to develop the site (Wave, 1948) and in October, site preparation had begun (Long Island Star-Journal, 1948e). The first phase of site preparation involved the creation of large sand dikes using hydraulic fill surrounding a portion of the planned 125-acre area in which waste disposal operations would be conducted. The sand dikes were constructed with periodic breaks to allow for drainage. The "bottom lands" were filled above the high water level with hydraulic fill and compacted (Long Island Star Journal, 1948c).

The waste filling activities were slated to begin on January 1, 1949, however, this was postponed for two-weeks to finish site preparations (Long Island Star-Journal, 1949a). Operations began adjacent to Cross Bay Boulevard and proceeded west to 83rd street. The next phase of waste filling started at the northern limit of 163rd Street at 83rd street and was advanced counterclockwise. The original 125-acre area proposed for filling was bounded by the intersection of 163rd Street and 83rd Street in the north, south along 83rd Street, east along 165th Street, and south along Cross Bay Boulevard and along the sand dikes. The shape of the southern portion of the SCP shoreline is entirely man-made. According to the 1949 Department of Sanitation Annual Report, in the first year, 30 acres of the Site were filled. Subsequent Annual Reports were not available to track the progress of filling over time. Opinion pieces from the time indicate the improved operations resulted in a dump that was not offensive to the community (Long Island Star-Journal, 1949b; Wave, 1949a). **Figure 2-4** shows the status of waste filling operations at SCP in 1951 and documents the man-made nature of the semi-circular shoreline along with the creation of land from open water.

Documents indicate waste disposal was proposed to occur in three phases (Long Island Star-Journal, 1948c). The 75-acre section at Cross Bay Boulevard and 165th Avenue was to be completed first and within 1 year, and the second and third stages were to include filling the parts of the site under water. Sand dikes for the remaining portion of the 125-acre site were prepared in 1949 after the Board of

Estimates approved additional funds (Wave, 1949b). Aerial imagery suggests filling proceeded as planned and in line with the proposed schedule. The Site reportedly only received waste from Queens (NYT, 1948), as opposed to previous operations that received waste from other boroughs. However, a strike in 1953 resulted in waste from Brooklyn being diverted to the Site, including incinerator residue (Long Island Star Journal, 1953). It is unclear how long the diverting lasted.

The 3-year time frame proposed for waste disposal operations at SCP was to coincide with the construction of an incinerator. However, records suggested that waste disposal may need to continue to support the disposal of incinerator waste (Long Island Star-Journal, 1948a). The promised incinerator, South Shore Incinerator, opened in 1954 (NYT, 1954). The opening of the incinerator coincides with around the timeframe in which waste disposal operations began to extend into the northern portion of the Site, beyond the originally planned 125 acres.

In 1951, additional filling was proposed for the Kissena Corridor Park in Queens to develop more parkland. However, that plan was abandoned in favor of expanding operations at the Site, given how well it was being operated (Long Island Star-Journal, 1951). The decision to abandon additional dumping at Kissena in favor of expanding operations at SCP supports the observations from aerial imagery that filling was extended beyond the initial 125-acres.

Based on aerial imagery and historical documentation, as waste disposal operations ceased in the southern portion of SCP, operations moved north. Aerial imagery and shoreline differences between the areas north and south of the Cove suggest the northern portion was not prepared with sand dikes in the same manner as the southern portion. There are fewer records documenting operations in the northern portion of the Site. The Phase 1 Archeological Study indicated ash was deposited primarily in the northern portion of the Site, which was supported by ash found in archeological soil borings conducted in this area (HDR, 2016). Waste disposal operations in the northern portion of SCP appear to start in 1953. Based on the 1954 aerial imagery, by 1954 the southern portion was completely filled and operations had begun in the north (**Figure 2-4**).

Photo documentation by EPA depicts Site operations and shows that electrical infrastructure, including power lines, was present at the Site (**Figure 2-5**). The use of a soil amendment containing sewage sludge as a source for artificial topsoil was promoted as an innovative, cost-saving solution to the problem of finding natural topsoil (NYCDP, 1950). A 1950 Memo from NYCDP, describes the process in detail and supports the designation of the southern portion of the Site as having been created using waste fill (**Figure 2-6**).

Aerial imagery from 1961 suggests that the entire Site was filled and that the Site was being covered with the soil amendment containing sewage sludge and sand (**Figure 2-7**). One record suggests filling was complete by 1957 (New York World-Telegram and Sun, 1960). An additional record indicates that over six years, three million tons of refuse was dumped at the Site (Long Island Star-Journal, 1962).

The process of creating the soil amendment using sewage sludge was dubbed “Operation Sludge” (Brooklyn Daily Eagle, 1953). Sludge from wastewater treatment plants was transported to sites and sprayed across drying beds. 16 layers were required to create 4 inches of dried sludge. The dried sewage sludge layer was mixed with the existing sand cover to create seven inches of sludge sand that was then mixed with three inches of clay to create nine inches of topsoil (NYCDP, 1959) (**Figure 2-6**). Rye grass was first planted and then replaced by different plants and trees (NYT, 1955). The sources of the clay

used for the topsoil were noted as "contracted", but their exact origin is unknown. (Brooklyn Daily Eagle, 1953)

At SCP, numerous sludge drying beds were created and are visible on aerial photographs from 1961 and 1966 (**Figure 2-7**). One documented source of sludge was the WWTP at Idlewild airport. A pipeline was built from the WWTP to the Site to convey the sludge to the drying beds (Long Island Star-Journal, 1962). Based on aerial imagery showing remnants of the sludge drying beds at various stages of fading, the creation of the soil amendment appears to have followed a sequence similar to the progression of filling, with the soil amendment first being placed in the southeast portion of the Site near Cross Bay Boulevard and then extending west and north. Aerial imagery can positively confirm certain areas in use for sludge drying beds at the time of the photograph. However, faint rectilinear lines on the aerial photos suggest that other areas may have also been used as sludge drying beds. These lines suggest sludge was produced across most of the southern portion of the Site and extended into the northern portion of the Site up to approximately the intersection of 161st Avenue and 78th Street. Sludge production may have extended further north, but there is no confirmatory documentation. However, it is likely that the soil amendment containing sewage sludge was placed over the entire Site. It is unclear when sludge management activities ceased at the Site, but aerial imagery suggests it was sometime between 1966 and 1975 (**Figure 2-7 and Figure 2-8**).

Aerial imagery from 1951 (**Figure 2-4**) shows a staging area for vehicles at the Site south of the intersection of 165th Avenue and 92nd Street. Support structures, presumably to facilitate the waste disposal and soil amendment operations at the Site, are first visible in the 1954 aerial imagery with several small buildings located just west of the western end of 165th Avenue. Aerial imagery from 1961 shows one larger building in the same general area as the previous smaller buildings. The structures are present in the 1966 imagery but are gone by 1975 (**Figure 2-7 and Figure 2-8**).

After Waste Disposal Operations (Post 1960s)

The historical records reviewed provided minimal information on any formal operations at the Site after waste disposal operations ended. When plans for the "Super Dump" were first developed, Robert Moses stated, "…a shorefront picnic park can be constructed in the area. This park will contain a parking field, concession buildings, fireplaces, picnic benches and tables and will be similar to the Plumb Beach Park, the cost of this development will be $400,000 (Long Island Star-Journal, 1948a)." The proposed recreation facilities at SCP were never realized.

In the early 1970's, the Board of Estimates proposed a study for plans for additional waste disposal at SCP. The area of SCP had earlier been dropped from plans to include it in the Gateway (Leader-Observer, 1973). However, civic opposition and coordinated efforts from politicians resulted in the Board of Estimates vetoing the plan for additional waste disposal operations at SCP. Ultimately, the City Planning Commission supported the initiative to include SCP in the Gateway plan (Leader-Observer, 1973). In 1974, ownership of SCP transferred to the Unites States Government as part of the deed to establish Gateway for use and development by the NPS (New York State, 1974).

Following transfer of ownership, NPS completed numerous assessments of the newly acquired resources of the Gateway. Assessments included:

- A detailed topographical survey which included the areas of SCP (Lockwood, Kessler & Bartlett, 1974)
- A general history of the Jamaica Bay, Breezy Point, and Staten Island Units (Wren, 1975). The general history makes no specific mention of SCP.
- A Historic Resource Study, Jamaica Bay: A history (Black, 1981). In relationship to the Site, the study mentions that, "The most dramatic changes in this area consisted of the post-World War II Landfill Projects at Spring Creek and Pennsylvania Avenues. (p.80)"
- An inventory map of key structures (NPS, 1975). No structures are identified at SCP.

In 1978, NPS developed and implemented plans for a guard rail along the street-side perimeter of the park. The guard rail allowed for access at three drive gates located at 159th Avenue, 164th Avenue and 92nd Street. (NPS, 1978). The guard rail is still present at the Site as of 2021. However, in many places it is not visible due to tall vegetation. Between 165th Avenue and 164th Avenue presumably adjacent residents have installed a wooden fence and have landscaped the area between the fence and the street. Other areas between the guard rail and street have undergone minor alterations such as paving or mowing.

NPS developed a General Management Plan (GMP) in 1979 for Gateway that zoned SCP "to maintain or enhance their use as "community park" while permitting the necessary protection of the fragile bay fringes (NPS, 1979)." **Figure 2-9** shows an illustration of proposed 1979 GMP land use areas within SCP. The zoning protected sensitive marsh edges while permitting management of the remainder of the Site as a community park. A natural shoal area along the south was to be renovated as a beach. Within the Site, there would be structured recreation areas as well as unstructured recreation areas with landscaped open spaces. Additionally, three support zones were designated to permit establishment of adequate facilities to support the planned use (NPS, 1979). To date, no significant recreational development has been undertaken at SCP.

In 2014, NPS issued an updated General Management Plan and Environmental Impact Statement (GMP/EIS) for Gateway (NPS, 2014b). The 2014 GMP/EIS favored alternative for management was "Alternative B: Discovering Gateway", which proposes that the eastern portions of the Site bordering adjacent roadways be used for recreation and that the remainder of the Site be maintained and improved as natural areas. The plan suggests in the recreation zone new facilities such as trailheads and parking areas, orientation kiosk, trails, and picnic areas would be developed to invite recreational use and promote exploration of the SCP area. In the natural zone, efforts to control *Phragmites* would be increased, saltmarsh and forested areas would be monitored, assessed, restored, protected and maintained to promote resiliency, social trails would be eliminated. Within the natural zone, access would be limited to designated trails and water access would also be developed, such as boat launch and landing sites, observation decks, and fishing access areas (NPS, 2014b).

SCP is noted as a place where the following changes may occur:

Desired Experience	Types of Change
Physical and programmatic connections created to link Gateway sites, New York City parks, and neighborhoods to Jamaica Bay	• Explore new linkages by trails, sidewalks, paths and bridges • Work with NYC and other partners to introduce new recreational skills and educational and interpretive programs in adjacent parks and communities
Orientation portals established to provide information on sites and activities throughout all Jamaica Bay park lands	• Create distinctive access corridors to parks and Jamaica Bay through art, signs and other visual expression • Improve signs and wayfinding • Use virtual and modern technology for orientation and maps
Recreation improvements	• New picnic and open space area

Figures 2-9 and 2-13 illustrate the 2014 GMP management zones for SCP. Future development at SCP is subject to the 2014 GMP/EIS.

The Site is currently used for active and passive recreation with fishing and hiking (USACE, 2017a) and evidence of homeless encampments has also been observed at SCP. However, thick vegetation conditions limit and/or discourage public use in most areas of the Site. There are no formal improvements or buildings on the Site to support recreation. The Site contains approximately 3 miles of fire roads that allow for pedestrian access (**Figure 1-2**). Illegal all-terrain vehicles also drive along the fire roads and unmaintained trails across the Site. Present-day illegal littering and dumping also occurs at the Site as evidence by accumulated trash visible at the surface in areas.

Analyzing aerial imagery from 1975 through 2021 indicates an evolution of unmaintained trails at the Site over time (**Figures 2-8 and 2-10**). From 1975 to present, the primary historical roads used to access the Site for historical operations, that later transitioned into portions of the fire roads, remain the most visible routes through the Site.

Several initiatives have proposed and advanced plans for ecological restoration at SCP. SCP was first recommended as a potential restoration opportunity through the USACE's Jamaica Bay, Marine Park, and Plumb Beach Ecosystem Restoration Feasibility Study which was initiated in 1996 (USACE, 2013). This initial feasibility study proposed restoration of 151.6 acres of habitat including 49 acres of low marsh, 10 acres of high marsh, and 6 acres of tidal creek (**Figure 2-11**) (USACE, 2017a).

Following Super Storm Sandy in 2012, the New York State Department of Environmental Conservation (NYSDEC) was awarded a grant from the FEMA HMGP for hazard mitigation at SCP (USACE, 2017a). As part of the HMGP, NYSDEC, USACE, and NPS reevaluated the restoration plans to include NNBFs to also provide CSRM benefits and enhanced coastal resiliency to the Howard Beach Community (USACE, 2016). These efforts were also coordinated with the Governor's Office of Storm Recovery's Howard Beach New York Rising Community Reconstruction Plan (Howard Beach Planning Committee, 2013).

The revised conceptual design for restoration included "natural and nature-based features including reuse of sandy dredged material; restoration of coastal and freshwater wetlands, maritime grassland, shrub, and forest plant communities; and construction of a vegetated berm to reduce the risk of flooding to the

adjacent neighborhood" (Princeton Hydro, 2021). Additional design efforts by Scape Studio envisioned a connector pathway running along the berm to incorporate an immersive landscape trail (Scape Studio, 2021). **Figure 2-11** illustrates the revised concept plan as well as design imagery of the proposed connector pathway.

The Jamaica Bay, Marine Park, and Plumb Beach Ecosystem Restoration Feasibility Study along with five other feasibility studies for different areas were combined within the Hudson-Raritan Estuary (HRE) Ecosystem Restoration Feasibility Study Final Integrated Feasibility Report & Environmental Assessment Report intended to streamline parallel efforts and maximize efficiencies, resources, and benefits (USACE, 2020). The SCP project was screened out because the Site was already being advanced under the FEMA Hazard Mitigation Grant Program. However, in 2019, FEMA decided not to fund the HMGP project (JCO, 2020). As such, the April 2020 Final HRE Ecosystem Restoration Feasibility Study Final Integrated Feasibility Report & Environmental Assessment Report acknowledges that the Site is no longer moving forward under the HMGP and has now been requested to be the first new phase feasibility study spin-off using the HRE authority (USACE, 2020).

Fires at the Site have been noted over time and continue to be a management priority. In 1971, prior to ownership conveyance, a newspaper article mentions that "Recently, many community leaders have complained that frequent fires have erupted at this landfill, believing it was due to the inadequate covering of the landfill (Leader-Observer, 1971)." The phragmites monoculture at the Site has been attributed to the periodic fires which keep the area in that successional stage (JBEC, 1984). A significant refuse fire on April 27, 1989 was documented by NPS as having occurred in the northern portion of the Site (NPS, 1989). Another brush fire burned approximately 7 acres in the southern portion of the Site on April 6, 2019 (Tidewater, 2019). The most recent conceptual designs for restoration included specific considerations for Fire and Emergency Access and a firebreak as an edge treatment to protect the adjacent neighborhood (USACE, 2017b).

In 2018-2019, NPS accepted 77,000 cubic yards of sand donated by a private entity which was stockpiled in the southern portion of the Site (JCO, 2020).

2.1.3 Waste Characteristics

Waste fill is defined as material derived from New York City Department of Sanitation's historical waste disposal operations at the Site. The waste fill includes refuse (heterogeneous mixture of municipal and industrial wastes and excavation and construction materials), incinerator residue (incinerated waste material), and coal ash from coal fired boilers/furnaces. The waste fill boundary encompasses most of the Site.

Past soil borings at the Site have identified the following in waste fill: glass, plastic, tile, brick, newspapers, rubbish, wood, processed wood, porcelain, netting, roof shingles, fiberglass insulation, plaster, fibers, rubber, paint chips, cardboard, coal, ash, concrete, cinders/slag (i.e., from waste incineration), bolts, metal, wire insulation, tires.

Sewage sludge was used as a component of a soil amendment (artificial topsoil) applied over waste fill to facilitate vegetative growth. The radiological artifacts present at the Site were found within the waste fill boundary of the Site.

2.1.4 Site Geology and Hydrogeology

The Site is situated in the Northern Atlantic Coastal Plain Physiographic Province comprised of seaward (to the east and southeast) thickening wedge of unconsolidated sediments overlying crystalline bedrock. This section describes the underlying geology from the surficial soil to the underlying unconsolidated sediments to the consolidated bedrock at a depth of 600 feet (ft) to 700 ft below ground surface (bgs). The buried bedrock surface slopes to the southeast.

The surficial geology of the Site primarily consists of artificial fill comprised of waste fill, hydraulic fill, discrete areas of construction and demolition debris, and the soil amendment containing sewage sludge mixed with clay. The majority of the Site is mapped as the Gravesend and Oldmills (GOB) soil unit comprised of coarse sand underlying 0 to 8 percent slopes (National Resource Conservation Service 2021). The soil composition is consistent with the Site being covered with artificial soil comprised of sewage sludge and lesser amounts of clay.

Underlying the fill material is a thin veneer of Holocene deposits consisting of gray clay, silt with peat (marsh deposits) and with sand stringers (low gradient historical fluvial [stream] deposits) and nearshore sand deposits (Open Water Jamaica Bay). **Figure 2-1** shows the Site land area prior to filling and the shoreline and zones where likely fluvial deposits (stream), marsh (clay and peat), and open water (sand) deposits are present. NPS mapped and identified a rather continuous Holocene Deposit beneath the Site ranging in thickness from approximately 2 to 8 ft thick at elevations ranging from 5 ft to minus 10 ft below mean sea-level.

Underlying the Holocene deposits are Pleistocene glacial deposits consisting of the upper Pleistocene deposits (Qu), followed by the Gardners clay, and Jameco gravel. The upper Pleistocene deposit is approximately 100 ft thick and consists of glacial outwash comprised course to fine gravel with sand, The Gardners clay (Qg) is a thin interstadial glacial deposit and is less than 25 ft thick and occurs approximately 200 ft below sea level. Gardners clay and is underlain by the Jameco gravel (Qj) is approximately 50 ft thick. (Buxton and Shernoff, 1999)

The Pleistocene glacial deposit unconformably overlie (erosional contact) the Cretaceous deposits consisting of the Magothy Formation (Kmm), Raritan Formation (Krc) and Lloyd Sand Member (Krl) of the Raritan Formation. The Cretaceous deposits have a total approximate thickness of 175 ft thick.

Precambrian (age) bedrock unconformably underlies (erosional contact) the Raritan Formation. The bedrock is comprised of folded and faulted gneiss and schist (metamorphic rock) at depth of 600 to 700 ft bgs.

Underlying the Site, groundwater is present in two water bearing zones; one deep and confined or semi-confined (Magothy and Llyod Aquifers) and one shallow and unconfined (Jameco Aquifer). A layer of peat and clay separates the zones. Shallow groundwater in the area is generally less than 10 ft bgs and was encountered at less than 5 ft bgs in monitoring wells bordering the neighborhood. Shallow groundwater generally flows from the inland edges of the Site towards the coastline (i.e., away from the adjacent neighborhood). Deep groundwater, is tidally influenced with deep groundwater flowing toward the coastline during low tides and inland during high tides. Deep groundwater is therefore thought to be a mix of both inland freshwater and saline coastal inputs. (JCO, 2020) No drinking water wells are located within the Site boundaries or downgradient with respect to groundwater flow direction (NPS, 2017).

2.1.5 Site Hydrology

Site surface water features include a small wetland area near the Cove, a small shallow pond near the northeast- corner of the Site, and a drainage ditch along the northern edge of the Site (JCO, 2020). The United States Fish and Wildlife Service (USFWS) National Wetlands Inventory (NWI) shows two additional small, isolated wetland areas in the southern portion of the Site; however, field verification indicated the identified areas do not exhibit wetland characteristics.

The drainage ditch along the northern edge of the Site is roughly 500ft in length and receives runoff from the area south of the Belt Parkway and higher elevation areas within the Site. The adjacent roadway to the east (78th Street) appears graded in manner that directs surface runoff toward the curb and inlets into the storm sewer. Stormwater runoff from the adjacent residential development is carried via a storm sewer system east to outfalls in Shellbank Basin (New York City Department of Environmental Protection [NYCDEP], 2021). The flow rate and hydraulic condition of the drainage ditch are unknown. The ditch discharges to Old Mill Creek and Jamaica Bay. The drainage ditch is likely locally tidally influenced near the discharge point.

The Site is mostly pervious with several miles of compacted fire roads. Surface runoff follows topography and generally slopes from the eastern boundary of the Site towards the Old Mill Creek and Jamaica Bay (**Figure 1-2**). Historical on-Site streams, including Crum Hill Creek that had its outfall to Jamaica Bay at the location of the Cove and an unnamed stream in the northern portion of the Site, no longer exist. There are six, 2.5 ft diameter pipe outfalls near the Cove (JCO, 2020). The upgradient pipe layout and purpose of the pipe outfalls is unknown but assumed to be related to channelization and subsequent burial (using pipes) of the historical Crum Hill Creek.

Contaminants within sediment, surface and subsurface soil may be transported to Site surface water through erosion from surface runoff and sloughing of surface and subsurface soil into the Site surface water features.

The major marine surface water features in the vicinity of the Site include the Jamaica Bay. Major freshwater surface water features in the vicinity of the Site include Ralph Creek and Spring Creek located north of the Belt Parkway. Spring Creek and Ralph Creek combine to form Old Mill Creek which passes under the Belt Parkway and also separates the Site from the nearby Pennsylvania Avenue Landfill, now Shirley Chisholm Park. The western boundary of the northern portion of the Site forms the eastern bank of the Old Mill Creek that flows into Jamaica Bay. The Spring Creek wastewater treatment plant which holds and treats Combined Sewer Overflow (CSO) flows is located just north of the Belt Parkway and outflows to Old Mill Creek.

2.1.6 Local Climate

Under the Köppen climate classification, the SCP Site is within a humid subtropical climate zone (Cfa), surrounded by greater NYC which is classified as humid continental (Dfa) (PlantMaps, 2021). Queens, NY experiences a strong annual temperature cycle, characterized by cold winters and warm summers (JCO, 2020). According to the closest National Oceanic and Atmospheric Administration (NOAA) weather station at John F. Kennedy Airport, the average seasonal temperature ranges are as follows (NOAA, 2021):

- Winter (Dec-Feb): 28.6-41.6°F
- Spring (Mar-May): 43.5-58.8°F
- Summer (Jun-Aug): 66.4-81.1°F
- Fall (Sept-Nov): 50.4-64.7°F

Average annual precipitation is 43 inches (NOAA, 2021). Monthly precipitation ranges from around 2.6 to 4 inches and is fairly evenly spread throughout the year. Snowfall is most common December through March with February having the highest average snowfall (NOAA, 2019).

2.1.7 Sensitive Environments

The Site has several sensitive areas that could be affected by environmental contaminants. These areas include tidal marshes, coastal areas, wetlands, and adjacent waters of the Jamaica Bay. There are no NPS designated Sensitive Resource Subzones at the Site (NPS, 2014b). The Site is open to the public. However, thick vegetation conditions dominated by *Phragmites* limit and/or discourage public use in most areas of the Site.

2.2 Summary of Previous Investigations

Past investigations of the Site were driven primarily by initiatives for restoration and hazard mitigation at SCP. As such, the investigations were designed specifically to meet the objectives of the restoration and hazard mitigation designs and investigate potential risks remaining after the proposed project implementation. The exposure scenarios were tailored to the proposed project implementation. The information gathered from past investigations was used to inform the current CSM and identify remaining data gaps for exposure scenarios aligned with the Gateway 2014 GMP/EIS (**Section 2.4**).

Past investigations and evaluations of the Site include, in chronological order:

- 2002 Hazardous, Toxic, and Radiological Waste (HTRW) Investigation (AMEC, 2002)
- 2016 Phase 1 Archeological Sensitivity Assessment (HDR, 2016)
- 2017 SI and SLERA (USACE, 2017a)
- 2017 Limited Gamma Radiation walkover survey (USACE, 2018)
- 2017 RSE Report (NPS, 2017)
- 2020 EE/CA Field Investigation (JCO, 2020)

Each investigation or evaluation and results are described below. Historical gamma survey coverage is presented in **Figure 2-12** and boring, sampling, and removal locations from these investigations are shown in **Figure 2-13**. **Appendix A** presents relevant figures from previous investigations performed at the Site.

2.2.1 AMEC 2002 HTRW Investigation (AMEC, 2002)

The 2002 HTRW sampling program at SCP was advanced as part of the USACE New York District feasibility level study of the Jamaica Bay Ecosystem Restoration Project. SCP was one of several

proposed restoration sites that was selected for screening relative to the presence of HTRW. Sixteen (16) subsurface soil samples were collected from 10 unique locations. Four composite hand augured samples from surface to a maximum of 2.5 ft bgs were collected from four locations all within the northern portion of the Site and near the shoreline. Six discrete Geoprobe® samples were collected at depths ranging from 3.8-8 ft bgs at six locations, two in the northern portion (one along the shoreline, one slightly inland) and four in the southern portion of the Site all near the shoreline. In addition to the discrete samples, composite samples from 8-12 ft bgs were collected from each of six Geoprobe® locations. The locations for sampling were mostly near shoreline areas likely because the original restoration design focused on creating coastal marshlands in the north and coastal dunes in the south of the Site. All soil samples were analyzed for volatile organic compounds (VOCs), semi-volatile organic compound (SVOCs), pesticides, polychlorinated biphenyl (PCBs), total petroleum hydrocarbons (TPH), Priority Pollutant Metals (13), dioxin and furan (2,3,7,8-TCDD and 2,3,7,8-TCDF), and Total Organic Carbon (TOC). The six discrete soil samples were also analyzed for grain size distribution.

Based on borings, fill materials was encountered at depths up to 12.5 ft bgs. No VOCs or pesticides were detected above NYSDEC Recommended Soil Cleanup Objectives (RSCO) based on the January 1994 Division Technical Administrative Guidance Memorandum (TAGM) on Determination of Soil Cleanup Objectives and Cleanup Levels (HWT-94-4046). Two SVOC detections (one each benzo(a)pyrene and dibenz(a,h)anthracene) exceeded RSCOs. Four PCB detections (one each Aroclor 1232, Aroclor 1248, Aroclor 1254, and Aroclor 1262) exceeded RSCOs. Arsenic, beryllium, cadmium, chromium, copper, lead, mercury, nickel, and zinc were detected at concentrations in excess of RSCOs, and two lead detections also exceeded the Eastern United States background concentration range. No values exceeded hazardous-waste thresholds. Comparing polycyclic aromatic hydrocarbons (PAHs) with New Jersey Department of Environmental Protection (NJDEP) historical fill values, the mean PAHs values at SCP were two orders of magnitude less than the historical fill value means. Notably historical fill material does not include municipal solid waste landfill sites (AMEC, 2002).

2.2.2 HDR 2016 Phase 1 Archeological Sensitivity Assessment (HDR, 2016)

The 2016 Phase 1 Archeological Sensitivity Assessment was carried out to advance the Spring Creek Coastal Storm Risk Management Project. Site investigations associated with the assessment included 15 geotechnical borings reaching up to 50 ft bgs. Geotechnical borings included six in the northern portion (three along the coast and three inland), one near the Cove and eight in the southern section (four along the shoreline and four inland). Locations were selected based on specific information required for the construction of the NNBF risk management features within specific habitat types of wetland, marine forest, or berm.

No analytical samples were collected from the geotechnical borings. The archeological sensitivity assessment involved an archeological survey comprised of a literature review and analysis of soil boring and an architectural survey. In addition to the 15 geotechnical borings, the assessment also incorporated soil boring results from the 2002 HTRW investigation and the 2017 SI and SLERA. The assessment determined that the proposed project activities would not adversely affect any potential subsurface archeological deposits, historical buildings, or structures. The report includes useful precontact and historical period summaries. Notably, the historical period summary states, “By the 1950s, the marshlands composing the Project area, already subject to dredging, landfilling and dumping activity from the turn of

the century, became an incinerator ash dumping ground for the DSNY, with the northern sector of the Project area a locus of incinerator refuse activity (HDR, 2016)."

2.2.3 USACE 2017 SI and SLERA (USACE, 2017a)

The 2017 SI and SLERA was carried out to advance the Spring Creek Coastal Storm Risk Management Project. The investigation characterized soil and sediment chemistry to determine acceptability of the proposed designs. Results were to inform modification to the grading and plan and assess ability for excavated material to be managed on Site or if offsite disposal would be needed.

The site investigation activities involved advancing a total of 388 soil borings with 5 borings in each of 75 one-acre grid cells, plus 13 borings surrounding the location SC-GP-1 from the 2002 HTRW sampling that identified elevated PCB levels within the area proposed for excavation. Two composite samples were created from the 5 borings within each of the 75 one-acre grid cells. One Group A sample was composited from soil from the surface to 2 ft above the designated depth for each boring determined by the proposed excavation depth at that location. Group A samples represented the "Cut Layer". One Group B sample was composited from soil from 2 ft above the designated depth for each boring to the bottom of the boring. Group B samples represented the "Exposed Layer". In total, 75 Group A samples and 75 Group B samples were analyzed. The one-acre grid cells were located in areas where the greatest thickness of material was proposed to be excavated. Of the 75 grid cells, 37 grid cells were along the northern portion shoreline, 34 grid cells were along the southern portion shorelines, and four inland grid cells were near the western end of 165th Avenue and 164th Avenue. Group A and Group B samples were analyzed for SVOCs, pesticides, PCBs and Priority Pollutant Metals and barium. Contaminants identified above NYSDEC Part 375 SCOs included PAHs, pesticides, PCBs, and numerous metals.

The 13 borings surrounding the location of SC-GP-1 from the 2002 HTRW sampling were intended to delineate the extent of the area of elevated PCBs. Borings were advanced to 14 ft bgs at 10, 25, and, 50 ft offsets north, south, east and west of a central borehole co-located in the vicinity of SC-GP-1. Seven composite samples were collected from each boring from two-foot intervals for a total of 91 samples. The PCB delineation samples were analyzed for PCBs and percent solids. PCBs exceeded the NYSDEC Unrestricted Use SCOs in 49 of the 91 samples with two samples exceeding 10 milligrams per kilogram (mg/kg) (one with 13 mg/kg PCBs and one with 260 mg/kg PCBs). The 260 mg/kg PCBs result at location PCB-E10 exceeded the 50 mg/kg limit indicating material subject to Toxic Substance Control Act (TSCA) regulation is on Site. Additional delineation to the north and south of PCB-E10 would be needed to delineate the extent of TSCA material. Additional borings east and west of the PCB-E10 would potentially reduce the extent of delineated TSCA material. A second area near sampling location SC-GP-4 from the 2002 HTRW with elevated PCB concentrations was not further delineated since no excavation was planned in that area.

2.2.4 USACE 2017 Limited Gamma Radiation Walkover Survey (USACE, 2018)

The 2017 limited gamma radiation walkover survey was implemented to inform ongoing CERCLA investigations at SCP and as a prudently conservative public safety measure to evaluate radiation levels on and near the fire roads. This survey was initiated in part due to radionuclide contaminants from waste fill present at GKP and the similarity in filling operations that took place at GKP to those that took place

at SCP. The surveys were focused on, and limited to, the fire roads and areas in the immediate vicinity. The surveys identified several locations where gamma radiation was in excess of the ambient levels and five man-made radioactive artifacts were removed from four unique locations. Three of the removal locations were along an east-west portion of the fire road through the middle of the southern portion of the Site. The fourth location was toward the northern boundary of the northern portion of the Site. Artifacts were recovered between 3 to 12" bgs.

2.2.5 NPS 2017 Removal Site Evaluation Report (NPS, 2017)

The 2017 Removal Site Evaluation (RSE) Report was carried out to assess the potential threat posed by the release or threatened release of hazardous substances to public health, welfare, and the environment, determine the need for additional CERCLA action, and evaluate whether a time-critical or non-time-critical removal action is appropriate. The RSE was based on readily available information, largely reports of prior environmental investigations. The RSE looked at CERCLA and NCP compliance, evaluated whether a release subject to the Clean Water Act has occurred, and evaluated the eight factors of NCP Section 300.415(B)(2) for determining the appropriateness of a removal action. Insufficient data was available to evaluate whether releases at the Site are affecting navigable waters or otherwise require a response under the Clean Water Act. Three of the eight factors of NCP Section 300.415(B)(2) support the decision to conduct a removal action at the Site; these include factors:

> (i) Actual or potential exposure to nearby human populations, animals, or the food chain from hazardous substances or pollutants or contaminants;
>
> (ii) Actual or potential contamination of drinking water supplies or sensitive ecosystems; and
>
> (v) Weather conditions that may cause hazardous substances or pollutants or contaminants to migrate or be released.

The RSE concluded that the release of hazardous substances at the Site potentially poses a threat to public health, welfare, and the environment, and therefore a removal action is appropriate. It was determined characterization of Site conditions, contamination, and associated risks is incomplete, and additional data collection is necessary to characterize certain Site conditions and support the selection of an appropriate removal action for the Site. It was concluded an EE/CA would be carried out for the Site to complete the requisite Site characterization, evaluate removal alternatives, and recommend an appropriate removal action for the Site.

2.2.6 NPS 2020 EE/CA Field Investigation (JCO, 2020)

The 2020 EE/CA Field Investigation was carried out to advance the Spring Creek Coastal Storm Risk Management Project. The objective of the investigation was to determine if concentrations of contaminants of potential concern in Site groundwater and surface water will pose an unacceptable potential for human and/or ecological risk after the implementation of the project.

The field investigations involved nine soil borings, including five borings in the northern portion of the Site and four borings in the southern portion of the Site. Five boring were located near the shoreline and

four were interior to the Site. A single discrete sample was collected from the soil borings at the water table or at an interval of obvious contamination (if present), except from NPS-B-03.

A total of 29 groundwater monitoring wells were installed at 21 unique locations, 12 locations were shallow only wells, one location was a deep only well, and eight locations were paired wells with one shallow well and one deep well co-located. Monitoring well locations were spread across the Site with 11 in the northern portion, two at the Cove, and 15 in the southern portion of the Site. Monitoring wells were located along the shoreline, interior of the Site, and inland.

Discrete soil samples were collected from each of the well installations in the same manner as from the soil borings, except from MW-20A. Soil samples were collected and analyzed to evaluate if fill materials were potentially acting as a source of contaminants of potential concern to groundwater and/or surface water. A total of 36 discrete subsurface soil samples were analyzed using synthetic precipitation leaching procedure (SPLP) for VOCs, SVOCs, pesticides, PCBS, dioxins and furans, and metals. Soil samples were also analyzed for radiological parameter gamma spectroscopy. Groundwater was sampled twice, once in 2018 and once in 2019. In both events, all groundwater samples were analyzed for VOCs, SVOCs, pesticides, PCBS, metals and radiological analytes, including radium-226, radium-228, total uranium, and gross alpha and beta. Dioxins and furans and water quality parameters of alkalinity, sulfide, dissolved organic carbon (DOC), chloride, sulfate and pH were analyzed for in six select wells and herbicides were analyzed for in 11 select wells. In 2018, two rounds of water level measurements were performed on all wells and within Jamaica Bay, one at low tide, and one at high tide. Additionally, in 2018, pressure transducers were installed in 11 wells and two locations in the bay for one full tidal cycle to collect groundwater level data to investigate tidal influence. Hydraulic conductivity testing was completed at seven wells in 2019.

Surface water samples were collected from eight locations. Two samples were collected from an on-Site wetland located in the middle of the Site, one sample was collected in a discrete pool of water in the northern corner of the Site, and two samples were collected from a drainage ditch along the northern edge of the Site. Five additional samples, considered reference samples, were collected from storm drains adjacent to and hydraulically upgradient of the Site. Surface water samples were analyzed for VOCs, SVOCs, pesticides, PCBs, metals (total and dissolved), dioxins and furans, hardness, radium-226, radium-228, total uranium, and gross alpha and beta.

Two radiological surveys were also conducted during the course of the field investigation. A limited gamma walkover survey was conducted in August 2018 over the area where sand donated to NPS by a private entity was later delivered to and stockpiled in the southern portion of the Site. One man-made radioactive artifact was located, removed, and disposed of at a licensed off-Site disposal facility. No additional specific sources were identified. Another limited gamma walkover survey was conducted in April 2019 over a 7-acre exposed area near Cross Bay Boulevard where a fire had occurred. Five areas with distributed, elevated radioactivity were identified.

Analytical results were compared with multiple screening criteria based on media type and human health and ecological risk which were the most recent screening levels as of 2020. VOCs were detected in multiple SPLP leachate samples and groundwater samples above screening levels. SVOCs were detected in multiple SPLP leachate samples, groundwater samples, and on-Site surface water samples above screening levels. Metals were widely detected in groundwater, surface water, and SPLP leachate samples.

Many metals were detected in groundwater and SPLP samples above screening levels. Metals in surface water were likely associated with suspended particles. Pesticides were detected above screening levels in groundwater and surface water samples. Herbicides were not detected in shallow groundwater above screening levels and were not analyzed for in any other medium. Two PCB Aroclors were detected in multiple SPLP leachate samples above screening levels, and only one groundwater sample contained PCBs above screening levels. PCBs were detected in three surface water samples above screening limits. Dioxins and furans were detected above screening levels in groundwater, surface water and SPLP leachate. However, dioxins and furans were also detected in the SPLP leachate method and SPLP leachate blanks and although the data was not rejected, qualifiers were applied to many of the SPLP leachate dioxin and furan sample results.

Following field work, but prior the completion of the report, FEMA decided not to fund the Coastal Storm Risk Management Project, also referred to as the HMGP project, which served as the basis for the exposure routes considered for the CSM at the time. The report thus recommended additional data collection via completion of a CERCLA RI under the remedial process to address data gaps, including characterization of the nature and extent of Site surface soil, such that potential human health and ecological risks associated with exposure to hazardous substances at the Site can be assessed and remedial alternatives necessary to address potential risks evaluated.

2.2.7 Data Quality / Usability

This section presents the usability of previously collected data from SCP as evaluated against the five USEPA general assessment factors (USEPA, 2003; 2012), including:

- **Soundness.** The extent to which the scientific and technical procedures, measures, methods, or models employed to generate the information is reasonable for, and consistent with, the intended application.
- **Applicability and Utility.** The extent to which the information is relevant for the project's intended use.
- **Clarity and Completeness.** The degree of clarity and completeness with which the data, assumptions, methods, quality assurance, sponsoring organizations, and analyses employed to generate the information are documented.
- **Uncertainty and Variability.** The extent to which the variability and uncertainty (quantitative and qualitative) in the information or the procedures, measures, methods, or models are evaluated and characterized.
- **Evaluation and Review.** The extent of independent verification, validation, and peer review of the information or of the procedures, measures, methods, or models.

Four of the six of the investigations summarized in **Section 2.2** involved the generation of analytical data. The following paragraphs provide a summary of the data usability for the historical investigations that involved sampling and analysis of environmental media.

AMEC 2002 HTRW Investigation: The 2002 AMEC HTRW Investigation involved the advancement of six soil borings and collection of 16 subsurface soil samples from 10 unique locations for chemical

analysis of VOCs, SVOCs, pesticides, PCBs, TPH, Priority Pollutant Metals (13), dioxin and furan (2,3,7,8-TCDD and 2,3,7,8-TCDF), and TOC. The data usability assessment is provided below:

Factor	Assessment
Soundness	The scientific and technical procedures documented in the report appear sound. The analytical data are approximately 20 years old and may not reflect present day analytical detection capabilities.
Applicability and Utility	Subsurface soil analytical data supports identification of Site contaminants.
Clarity and Completeness	Documentation is clear and complete. Standard QA/QC samples were collected. Soil boring logs and full laboratory reports are provided.
Uncertainty and Variability	The timeframe of the investigation (approximately 20 years ago) presents uncertainty regarding whether the data reflect current Site conditions.
Evaluation and Review	The data did not undergo independent verification or validation.
Conclusion: The 2002 AMEC HTRW Investigation provides limited, but reliable information regarding the depth, thickness, and presence of the waste filled area based on the soil borings. Due to age and lack of evaluation and review, the analytical data is usable for development of the CSM and to support planning for the RI but is not usable for remedial decision-making under CERCLA.	

USACE 2017 Site Investigation and Screening Level Ecological Risk Assessment: The 2017 USACE SI and SLERA involved advancing 388 soil borings and collection of 75 Group A and 75 Group B subsurface soil composite samples analyzed for SVOCs, pesticides, PCBs and Priority Pollutant Metals and barium and 91 subsurface samples analyzed for PCBs and percent solids. The data usability assessment is provided below:

Factor	Assessment
Soundness	The scientific and technical procedures documented in the report appear sound. The analytical data are six years old.
Applicability and Utility	Subsurface soil analytical data supports identification of Site contaminants. However, the composite samples are not directly comparable to discrete samples. Additionally, the composited sample depths were based on a grading plan associated with the HMGP Project as opposed to the Site CSM and/or current/future land use. Lastly, in many cases the soil borings were advanced to depths associated with the grading plan and did not fully delineate the vertical extent of waste fill. Borings and samples were also focused in areas planned for excavation and excluded much of the interior portions of the Site.
Clarity and Completeness	Documentation of analytical and sampling methods is clear and complete. Standard QA/QC samples collected. Soil boring logs and full laboratory reports are provided. However, the exact GPS coordinates of the completed soil borings are not available.
Uncertainty and Variability	The lack of GPS coordinates for the completed soil borings requires Geogrpaphic Information System (GIS) work using existing figures to approximate coordinates for each boring, which introduces uncertainty and variability. Specific details regarding the compositing procedure are also not provided.
Evaluation and Review	The analytical data underwent independent Level 4 data validation. The report underwent NPS and USACE review.
Conclusion: The 2017 USACE SI and SLERA provides limited information regarding the depth, thickness, and presence of the waste fill based on the soil borings. Due to low applicability, low utility, and high uncertainty, the analytical data is usable for development of the CSM and to support planning for the RI but is not usable for remedial decision-making under CERCLA.	

USACE 2017 Limited Gamma Radiation Walkover Survey: The USACE 2017 Limited Gamma Radiation Walkover Survey generated data of radiation levels for surface soil on and near the fire roads. The data usability assessment is provided below:

Factor	Assessment
Soundness	The scientific and technical procedures documented in the report appear sound and the survey was conducted using industry standard techniques for surveying radiological anomalies.
Applicability and Utility	The area surveyed is distributed across the Site and the data is relevant and applicable. The spacing of the survey tracks is fairly wide.
Clarity and Completeness	Documentation of survey methods is clear and complete. Instrument calibration records are provided. GPS coordinates of the recovered radiological items were not provided.
Uncertainty and Variability	The lack of GPS coordinates of the recovered radiological items requires GIS work using existing figures to approximate coordinates, which introduces uncertainty and variability.
Evaluation and Review	The report underwent USACE and NPS review.
Conclusion: Based on the assessment for many factors, the 2017 Limited Gamma Radiation Walkover Survey provides data that is usable for development of the CSM and to support planning for the RI but is not usable for remedial decision-making under CERCLA.	

NPS 2020 EE/CA Field Investigation: The 2020 EE/CA Field Investigation involved advancing 9 soil borings; installing 29 monitoring wells; collecting 36 discrete subsurface soil samples analyzed for SPLP - VOCs, SVOCs, pesticides, PCBs, dioxins and furans, metals and gamma spectroscopy; collecting two rounds of groundwater samples analyzed for VOCS, SVOCs, pesticides, PCBs, metals, radium-226, radium-228, total uranium, and gross alpha and beta, with select wells analyzed for dioxins and furans, herbicides and water quality parameters of alkalinity, sulfide, DOC, chloride, sulfate and pH; collecting surface water samples from 8 locations analyzed for VOCs, SVOCs, pesticides, PCBs, metals (total and dissolved), dioxins and furans, hardness, radium-226, radium-228, total uranium, and gross alpha and beta. The field investigation also documented two gamma walkover surveys and included hydraulic conductivity testing, water level measurements and tidal cycle water levels. The data usability assessment is provided below:

Factor	Assessment
Soundness	The scientific and technical procedures documented in the report appear sound. The analytical data are three years old.
Applicability and Utility	Groundwater and surface water are well characterized by this investigation. The subsurface soil sample analysis as SPLP limits the utility of subsurface soil data for the RI. The Gamma Walkover Survey data is relevant and applicable.
Clarity and Completeness	Documentation of analytical and sampling methods is clear and complete. Standard QA/QC samples were collected. Soil boring logs, full laboratory reports, and complete gamma walkover survey reading logs are provided.
Uncertainty and Variability	The report documentation is very thorough, and the investigation was carried out under an approved SAP.
Evaluation and Review	The analytical data underwent independent data validation with Stage 4 validation for the first data package for each media and Stage 2B validation for remaining data packages. The report underwent NPS review.

Conclusion: The 2020 EE/CA Field Investigation provides complete and useful information regarding the depth, thickness, and presence of the waste fill based on the soil borings and monitoring well installation logs. Based on the high levels of assessment for all factors, the analytical data is usable for development of the CSM, to support planning for the RI, and for remedial decision-making under CERCLA (i.e., the data is usable for the RI Report).

2.2.8 Preliminary Identification of Data Gaps

Based on the past investigations, the following data gaps were identified for SCP RI sampling:

- risk to human health and environment from Site-related chemical and radiological contaminants;
- nature and extent of chemical and radiological contaminants in surface soil (0-6 inches bgs), subsurface soil (below 6 inches bgs), and sediment;
- potential releases of chemical and radiological contaminants to soil and groundwater from operations in historical support areas;
- potential for VOCs detected in groundwater to be present in soil gas along the Site boundary;
- nature and extent of radiological anomalies and distributed contamination;
- potential presence of landfill gases resulting from waste fill; and
- reference area concentrations of chemical and radiological contaminants in soil and sediment.

SCP RI sampling has been designed to address these data gaps so that the results can be combined with the 2020 EE/CA Field Investigation to develop the SCP RI Report.

2.2.9 Contaminants of Potential Concern

Contamination is present at the Site due to historical waste disposal operations that resulted in the placement of waste fill and the soil amendment containing sewage sludge. Based on past investigations, contaminants of potential concern to be investigated through the SCP RI sampling include: VOCs, SVOCs including PAHs, pesticides, PCBs, dioxins/furans, metals, herbicides, and landfill gases.

Previous investigations have identified radiological contamination within the waste filled area at the Site. Based on the results of previous investigations, the radionuclides identified for further investigation are radium-226, uranium-238, and thorium-232.

2.2.10 Media of Potential Concern

The media of potential concern for SCP include:

- surface soil;
- subsurface soil;
- sediment;
- groundwater and;
- soil gas.

2.3 Current and Future Property Use Scenarios

Current Land Use. SCP is open to the public year-round. There are three gates to the park that prevent vehicle access but allow pedestrian access. The park is situated within a densely populated residential area and is used, according to local NPS workers, primarily for walking, jogging and/or birdwatching. There are several fire roads (mainly dirt or old pavement) that run throughout the park and down to the beach area; there are no playground areas. Some of the fire roads and unmaintained trails are also illegally used for all-terrain vehicles; there are also several social trails related to unauthorized use of the SCP, including fishing.

While visitors of the park will walk along the shoreline, people typically do not swim in the portions of Old Mill Creek or Jamaica Bay adjacent to the Site, according to conversations with NPS staff, although it is possible that some swimming may occasionally occur. Homeless encampments are routinely removed from SCP. Also, evidence of Hindu ritual practices along the shoreline of SCP near Cross Bay Boulevard have also been noted (HDR, 2016). Fishing (finfish or shellfish) has been observed to occur along the shoreline, although the New York State Department of Health (NYSDOH) has issued a fish consumption advisory for Old Mill Creek and Jamaica Bay due to the known presence of PCBs and other contaminants in these waterways. Recreational opportunities in the freshwater wetlands/streams in the park are limited by dense vegetation, shallow/intermittent water depths and unconsolidated substrate.

Coastal areas of the Site are characterized as stable sandy along the southern coast parallel to 165th Avenue and transitions to destabilized sandy around the lower half of the semi-circle of the southern portion (USACE, 2017). At the westernmost point of the southern portion of the Site, the shoreline transitions to marsh and continues as marsh to the northern Site boundary. The coastal areas and adjoining waters, the Old Mill Creek and Jamaica Bay, are directly adjacent to the Site where waste fill is present. Based on the coastal nature of SCP, Site groundwater is a mix of fresh water and saltwater. No water in or around SCP is currently being used for drinking water, which is not expected to change in the future.

Future Land Use. SCP is part of the Jamaica Bay Unit of Gateway. In June 2014, NPS issued the Record of Decision for the Final Gateway National Recreation Area General Management Plan/Environmental Impact Statement (Gateway GMP/EIS). Subsequent to public comment, NPS weighed the proposed alternatives and selected Alternative B: Discovering Gateway as the most appropriate for the future management of Gateway (NPS, 2014b).

Future land use at SCP is expected to remain primarily as a natural area with limited recreation, consistent with the management zones specified in the Final Gateway GMP/EIS. NPS intends to limit recreational use of SCP to only passive activities (trails, parking and picnicking areas) in the eastern portions of the park that border adjacent neighborhoods and roadways, and to restore and maintain the remaining portions of the park as natural areas. The plan suggests in the recreation zone new facilities such as trailheads and parking areas, orientation kiosk, trails and picnic areas would be developed to invite recreational use and promote exploration of the SCP area. In the natural zone, efforts to control *Phragmites* would be increased, saltmarsh and forested areas would be monitored, assessed, restored, protected and maintained to promote resiliency, social trails would be eliminated and access limited to designated trails, and water access would also be developed such as boat launch and landing sites, observation deck, and fishing access areas (NPS, 2014b).

Figure 2-14 presents a map of the Site showing the management zones specified in the Final Gateway GMP/EIS. As shown in **Figure 2-14**, a majority of the Site is specified as a natural management zone, with a limited recreation management zone along the eastern boundary of the Site adjacent to the residential area.

2.4 Graphical Conceptual Site Model

A graphical CSM of the Site is presented in **Figure 2-15** and the key CSM assumptions are discussed below.

2.4.1 Key CSM Assumptions

- Sources suspected to have contributed to Site-related contamination include areas containing waste fill, areas having received the soil amendment containing sewage sludge, and two historical support areas. Waste fill is present across a majority of the Site. Sewage sludge was dried in defined sludge pits and the soil amendment was spread widely across the Site. Two historical support areas existed at the Site.
- Radiological artifacts are items contained in waste fill. To date, the radiological artifacts identified at the Site emit gamma radiation in sufficient strength and frequency to enable their detection using traditional gamma walk over survey methodologies (i.e., when the source is located at or near the ground surface).
- The shallow Site geology consists of the soil amendment containing sewage sludge, overlying waste fill that was placed directly on top of the Holocene deposit or open water bay deposits. The Holocene deposit is a low permeability clay-silt lithology throughout most of the northern portion of the Site. The Holocene deposit and open water bay deposits overlies the Pleistocene glacial outwash, which is the primary water bearing interval at the Site.
- Underlying the Site, groundwater is present in two aquifers: (1) a shallow, unconfined aquifer that overlies the Holocene deposit and is in direct contact with waste fill and (2) the underlying regional aquifer within the Pleistocene glacial outwash deposit. The Holocene deposit serves as a confining to semi-confining layer between the two aquifers and there is a downward hydraulic gradient from the shallow aquifer to the regional aquifer. Hydraulic conductivities are variable between the shallow and deep aquifer and across the Site.
- Site contaminants present within the shallow aquifer may migrate through the Holocene deposit and into the regional aquifer in the Pleistocene glacial outwash deposit. Additionally, shallow groundwater flows from inland toward Jamaica Bay, with minimal tidal influence being observed in the shallow aquifer. The deep aquifer is tidally influenced flowing from inland towards Jamaica Bay at low tide and from Jamaica Bay towards the inland boundary during high tides.
- Surface water runoff is conveyed through the Site following the surface topography to the Jamaica Bay. Surface runoff from the northernmost area of the Site flows to a drainage ditch

which outfalls to Old Mill Creek. Surface runoff from the adjacent residential area is conveyed via storm sewers to Shellbank Basin.

- Subsurface soil contamination may be brought to the ground surface through bioturbation from plants and burrowing animals. Contaminants within surface and subsurface soil may be transported to Site surface water and sediment through erosion from surface runoff. Contaminants within surface and subsurface soil may be transported to groundwater via leaching and infiltration of precipitation.
- With respect to human and ecological receptors, the primary exposure media are the surface and subsurface soil, groundwater, surface water and sediment. Additionally, certain constituents may bioaccumulate into plants and animals, or may migrate into indoor air of a building via groundwater and soil gas. Exposure routes include dermal contact, ingestion, inhalation of dust and volatiles, and external radiation. **Figure 2-16** is a preliminary receptor pathway diagram that summarizes the sources of contaminated media, release mechanisms, contaminant transport, exposure media, and exposure routes to human and ecological receptors. The exposure routes summarized in **Figure 2-16** include the transport mechanisms and pathways that are considered complete, as well as those for which there are currently insufficient sample data to determine completeness. Analytical data and other information collected for the RI will be used to refine exposure media, exposure routes and receptors; based on these data, the preliminary receptor pathway diagram will be revised as needed to reflect the RI findings.

3 DQO Planning Team and Stakeholders

The DQO Planning Team includes the primary decision makers and project team members, such as risk assessors or remediation engineers, who will use the data generated as a result of the DQOs. The DQO Planning Team members, primary decision makers, and stakeholders for the RI are identified in **Sections 3.1, 3.2, and 3.3**, respectively.

3.1 Data Quality Objective Planning Team

The DQO Planning Team develops the project DQOs according to the DQO process. The DQO process is iterative, and team members may be added or changed to address technical issues that were not initially identified. The DQO Planning Team for the RI includes:

- Jeffrey Johnson, NPS, Environmental Compliance and Cleanup Division, Federal Government Lead (Jeffrey_G_Johnson@nps.gov)
- TBD, Cleanup Lead (TBD)
- Kathleen Cuzzolino, USACE, New York District (NAN), Project Manager (PM) (Kathleen.Cuzzolino@usace.army.mil)
- Marc Randrianarivelo, USACE, Baltimore District (NAB), Technical Manager (Marc.H.Randrianarivelo@usace.army.mil)
- Eric Barbour, NAB, Health Physicist (Eric.W.Barbour@usace.army.mil)
- Ryan Wensink, G2S, PM (ryan.wensink@tideh2o.net)
- Clif Gray, G2S, Project Radiological Lead (clif.gray@tideh2o.net)
- James Reese, G2S, Project Certified Health Physicist (james.reese@tideh2o.net)
- Sara McGarity, G2S, Project Engineer (sara.mcgarity@tideh2o.net)
- John Wyckoff, G2S, Project Geologist (john.wyckoff@tideh2o.net)
- Lisa McIntosh, Woodard & Curran, Risk Assessor (lmcintosh@woodardcurran.com)

3.2 Decision Makers

The decision makers have the ultimate authority for making final decisions based on the recommendations of the DQO Planning Team. The decision makers for this project are:

- Jeffrey Johnson, NPS, Environmental Compliance and Cleanup Division, Federal Government Lead
- Kathleen Cuzzolino, USACE, Cleanup Lead

3.3 Stakeholders

Stakeholders are parties who may be affected by the results of the investigation and/or persons who may later use the data resulting from the DQO process. Stakeholders for NPS-managed lands may include tribal governments, States, non-governmental organizations, and other Federal agencies. The stakeholders for this project, including those with jurisdictional or legal authority over the Site and/or areas affecting the Site, include:

- USEPA, Region II;
- NYSDEC;
- New York Department of Health (NYDOH); and
- U.S. Nuclear Regulatory Commission (USNRC).

4 Data Quality Objectives

The DQO process specifies anticipated project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified.

The DQO process consists of the following seven steps, described in detail below:

1. State the Problem;
2. Identify the Goal of the Investigation;
3. Identify the Information Inputs;
4. Define the Boundaries of the Investigation;
5. Develop the Analytic Approach;
6. Specify Performance or Acceptance Criteria; and
7. Develop the Plan for Obtaining the Data.

The following sections detail each step in the DQO process for this investigation.

4.1 State the Problem

Waste disposal operations were conducted at the Site from 1948 to the early 1960s. After the completion of waste disposal activities, sewage sludge was mixed with clay and used as a soil amendment to support revegetation. Historical investigations have been conducted to assess chemical and radiological contamination at the Site. These investigations identified the presence of man-made radiological artifacts (e.g., deck markers) buried in near surface soil and potential releases of radiological and chemical contamination to the environment. Based upon prior Site investigations and historical information, the potential contaminants include:

- metals (including mercury and hexavalent chromium)
- SVOCs/PAHs
- VOCs
- pesticides
- herbicides
- PCBs
- dioxins/furans
- radiological constituents (e.g., radium-226, thorium-232, natural uranium)

While there is a substantial amount of usable environmental data available for the Site, the current dataset is insufficient to complete the RI. Therefore, additional data are needed to characterize the nature and extent of contamination, evaluate the fate and transport of Site-related contamination, and determine if chemical and radiological contaminants are present at levels that pose an unacceptable risk to human and ecological receptors.

4.2 Identify the Goal of the Investigation

The primary goals of RI sampling are to:

- Further characterize the nature and extent of potential contamination at the Site, specifically in soil and sediment; and
- generate data of sufficient quality to complete the risk assessment.

Additional goals of RI sampling are to:

- determine whether contaminant releases to soil and groundwater occurred related to support activities in the 1951 and 1954 historical support areas;
- determine whether volatile contaminants detected in groundwater or soil leachate are also present in soil gas at the Site boundary;
- characterize concentrations of contaminants in soil and sediment that may be attributable to background sources, and
- assess whether landfill gases are present at levels that require further investigation.

While RI sampling has been designed to thoroughly characterize the Site, depending on the results, additional phases of investigation may be necessary to address data gaps and complete the RI.

4.2.1 Principal Investigation Question(s)

The principal investigation questions for the RI are as follows:

- Principal Investigation Question 1: Did support activities in historical support areas result in releases of chemical and radiological contamination to soil and groundwater? If so, what is the nature of contamination?

  Statement: Determine if contaminants concentrations in soil and groundwater in historical support areas indicate a release of chemical and/or radiological contamination that exceeds the PALs.

- Principal Investigation Question 2: What is the nature and extent of contamination in soil and sediment at the Site?

  Statement: Determine if contaminants are present in soil and sediment and evaluate their spatial distribution at the Site.

- Principal Investigation Question 3: Are VOCs that were detected in groundwater or soil leachate also present in soil gas above the vapor intrusion screening levels (VISLs) along the Site boundary?

  Statement: Determine if concentrations of VOCs are present in soil gas above the VISLs along the Site boundary.

- Principal Investigation Question 4: What is the nature and extent of radiological anomalies within the Site boundary?

Statement: Estimate the locations of near-surface radiological contamination as well as surface and subsurface anomalies in waste fill and evaluate representative locations to determine the source of elevated radioactivity.

- Principal Investigation Question 5: Are landfill gases present at levels that require mitigation?

  Statement: Determine whether landfill gases are present at levels that require mitigation.

- Principal Investigation Question 6: What are the concentrations of chemical and radiological contaminants in reference area soil and sediment?

  Statement: Estimate concentrations of chemical and radiological contaminants present in reference area soil and sediment.

4.2.2 Decision Criteria for Chemical Contamination

The following human health screening benchmarks will be used to evaluate the presence of chemical contamination:

- Surface Soil, Subsurface Soil, and Sediment – USEPA Regional Screening Levels (RSLs) (USEPA, 2021) for soil based on residential land use (residential criteria will be used to inform decisions regarding risk to human receptors);
- Groundwater – Federal Maximum Contaminant Levels (MCLs) for drinking water and USEPA RSLs for tap water (USEPA, 2021); and
- Soil Gas – USEPA Residential VISLs for target sub-slab and near-source soil gas.

Note: The USEPA RSLs correspond to a cancer risk of 1 x 10^{-6} and a hazard quotient [HQ] of 0.1 and represent the latest release at the time of writing the SAP (i.e., May 2021). The USEPA Residential VISLs correspond to a to a target cancer risk of 1 x 10^{-6} and a hazard quotient [HQ] of 0.1 and were retrieved from the USEPA VISL Calculator on October 22, 2021.

Chemicals that are detected at concentrations that exceed human health screening benchmarks will be identified as a contaminant of potential concern (COPC) for further evaluation in the human health risk assessment (HHRA) (see **Section 8**).

NPS Ecological Screening Values (ESVs) (NPS, 2018c) will be used as ecological screening benchmarks for surface soil and sediment. Chemicals that are detected at concentrations that exceed the relevant NPS ESVs will be identified as a contaminant of potential ecological concern (COPEC) for further evaluation in the screening level ecological risk assessment (SLERA) (see **Section 8**).

4.2.3 Decision Criteria for the Radiological Contamination

The following human health screening benchmarks will be used to evaluate the presence of radiological contamination:

- Surface Soil, Subsurface Soil, and Sediment – USNRC technical report designation (NUREG) 1757, Volume 1 Revision 2, Table B.2, Screening Values of Common Radionuclides for Soil Surface Contamination per 25 millirem [mrem] per year above background (USNRC, 2006) and

USEPA Radionuclide Preliminary Remediation Goals (PRGs) calculator (https://epa-prgs.ornl.gov/radionuclides/); and

- Groundwater – Federal MCLs for drinking water, including total radium (226/228) at 5 picocuries per liter [pCi/L], gross alpha of 15 pCi/L, gross beta of 4 mrem per year (50 pCi/L) and uranium of 30 µg/L. The methods selected to evaluate radiological contamination in groundwater and surface water align with the USEPA 2000 Radionuclide Rule, 66 Federal Register (FR) 76708, December 7, 2000, Effective Date December 8, 2003 (USEPA, 2000).

Radionuclides that are detected at concentrations that exceed human health screening benchmarks will be identified as a COPC for further evaluation in the HHRA and dose assessment.

NPS ESVs (NPS, 2018d) will be used as ecological screening benchmarks for surface soil and sediment. Radionuclides that are detected at concentrations that exceed the relevant NPS ESVs will be identified as COPECs for further evaluation in the SLERA and dose assessment.

4.3 Identify the Information Inputs

4.3.1 Previous Data Usability

Several environmental investigations have been conducted at SCP, primarily to advance ecological restoration initiatives (See **Section 2.2**). These investigations included sampling subsurface soil, groundwater, and surface water and radiological surveys with limited radiological removals, which provide useful information that was used to inform the Site CSM in **Figure 2-15**.

The analytical data produced during the 2020 EE/CA Field Investigation was validated in accordance with final, approved SAPs and the resulting data was determined to be usable for remedial decision-making under CERCLA (See **Section 2.2.1**). Additional data collection is needed because the existing usable data indicate the presence of radiological and chemical contamination, but do not provide sufficient spatial coverage in surface soil, subsurface soil, and sediment to adequately characterize Site conditions for remedial decision making. The results of the 2020 EE/CA investigation will be combined with the additional data collection efforts described in this SAP to support the development of the RI Report.

The following provides a summary of previous data that are usable in the RI:

- Groundwater data: The 2020 EE/CA Field Investigation produced groundwater data for 29 monitoring wells. Analytical data for VOCs, SVOCs, pesticides, PCBs, metals, radium-226, radium-228, total uranium, and gross alpha and beta, dioxins and furans, herbicides, alkalinity, sulfide, DOC, chloride, sulfate and pH will be included in the risk assessment.
- Surface water data: The 2020 EE/CA Field Investigation produced surface water data for eight locations. Analytical data for VOCs, SVOCs, pesticides, PCBs, metals (total and dissolved), dioxins and furans, hardness, radium-226, radium-228, total uranium, and gross alpha and beta activity will be included in the risk assessment.
- Radiological artifact removal data: Records of removals of radiological artifacts from the USACE 2017 Limited Gamma Radiation Walkover Survey and the two surveys documented in the 2020 EE/CA Field Investigation Report will be used to inform the answer to Principal

Investigation Question 4: What is the nature and extent of radiological anomalies within the Site boundary? **Table 4-1** provides a summary of radiological artifacts historically recovered from waste fill at the Site.

4.3.2 Data to be Collected in the Current Investigation

The investigation approach for the SCP RI has been designed to produce the data summarized in **Table 4-2**. The investigation approach is presented in **Figure 4-1** and the rationale for each specific sampling location is summarized in **Table 4-3** by sample media. **Section 5** details the field sampling approaches that will be used to obtain the data to be collected during the SCP RI. This approach has been designed to produce data to answer the principal investigation questions specified in **Section 4.2.1** and repeated for reference, as follows:

- Principal Investigation Question 1: Were there releases of chemical and/or radiological contamination to soil and groundwater from related support activities in historical support areas that exceed the PALs?
  - One discrete surface and one discrete subsurface soil sample will be collected in each of the historical support areas (see **Figure 4-1**). Historical support areas include the area south of 92nd Street shown in the 1951 aerial imagery (1951 support area) and the area west of 165th Avenue shown in the 1954 aerial imagery (1954 support area) (see **Figure 2-4**). Soil samples will be analyzed according to the analytical approach described in **Section 4.5**.
  - Groundwater samples will be collected from two new shallow monitoring wells (SCP-MW-22 and SCP-MW-23) to be installed within the historical support areas (See **Figure 4-1**). Groundwater samples will be analyzed according to the analytical approach described in **Section 4.5**.
- Principal Investigation Question 2: What is the nature and extent of chemical and radiological contamination in soil and sediment at the Site that exceed the PALs?
  - Surface and subsurface soil samples will be collected from 105 locations, which is based on achieving a minimum sample density of one soil sampling location per two acres within the waste filled area at the Site (See **Figure 4-1**). Subsurface soil cores will be advanced to 10 ft bgs at each sampling location. Soil samples will be analyzed according to the analytical approach described in **Section 4.5**, with 25% of surface soil samples being analyzed for TOC to support the risk assessment.
  - Sediment in areas of existing on-Site surface waters will be collected at the same locations as the on-Site surface water samples collected as part of the 2020 EE/CA investigation (See **Figure 4-1**). Sediment samples will be analyzed according to the analytical approach described in **Section 4.5**. All sediment samples will be analyzed for TOC to support the risk assessment.

- Principal Investigation Question 3: Are VOCs that were detected in groundwater or soil leachate also present in soil gas above the VISLs along the Site boundary?
  - Ten probes will be installed using direct push technology (DPT) to evaluate soil gas. The soil gas probe locations are presented in **Figure 4-1**. At each location, a boring will be advanced to a depth of 5 ft bgs and a soil gas probe will be constructed in the vadose zone with a screen placed between 4 and 5 ft bgs. A discrete soil gas sample will be collected from each probe using a summa canister. The soil gas samples will be analyzed for VOCs as indicated in the analytical approach described in **Section 4.5**.

- Principal Investigation Question 4: What is the nature and extent of radiological anomalies within the Site boundary?
  - A refined gamma walkover survey (GWS) will be performed of all fire roads (plus a 10 ft buffer on either side) (See **Figure 4-1**). The GWS will be performed to locate surface and near-surface radiological anomalies indicating the potential presence of radiological artifacts. An in-situ gamma spectroscopy system will be used to quantify the radionuclides in selected areas based on the results of GWS.
  - Intrusive investigations of representative anomalies or anomaly areas will be performed, including the collection of biased count rates, dose rates, and soil samples at the ground surface and on contact with the source of the anomaly. Soil samples to investigate radiological anomalies will be analyzed according to the analytical approach described in **Section 4.5**. In addition, physical measurements, a photolog, and a gamma spectrum will be generated for each radiological artifact or source of contamination recovered during focused investigations.
- Principal Investigation Question 5: Are landfill gases present along the Site boundary at levels that exceed the PALs?
  - At each of the 10 soil gas probe locations presented in **Figure 4-1**, field screening-level sampling/analyses will be conducted using a portable (direct reading) instrument (e.g., combustible gas indicator [GEM^{TM}2000 Plus with the targeted gas probes or similar]) to determine the presence/levels of methane (CH_4) (%), carbon dioxide (CO_2) (%), oxygen (O_2) (%), carbon monoxide (CO) (ppm), and hydrogen sulfide (H_2S) (ppm). The GEM^{TM}2000 Plus will also provide measurements of barometric pressure, and temperature at each soil gas probe. Due to the age of the landfill (i.e., over 60 years since operations ceased around 1960) and because an engineered cap was not installed (i.e., landfill gases would not accumulate), high levels of landfill gases are not anticipated. However, if field screening determines that methane is present above 5% in soil gas, then further evaluation may be necessary to fully characterize landfill gases throughout the Site.
- Principal Investigation Question 6: What are the concentrations of chemical and radiological contaminants in reference area soil and sediment?

- Surface and subsurface soil samples will be collected from 15 sampling locations within a reference area across the Belt Parkway from SCP (See **Figure 4-1**). Subsurface soil core depths will be to 10 ft bgs. Reference are soil samples will be analyzed according to the analytical approach described in **Section 4.5**.
- Sediment samples will be collected at five locations within a stream located within the established reference area across the Belt Parkway from SCP (See **Figure 4-1**). Reference area sediment samples will be analyzed according to the analytical approach described in **Section 4.5**.

4.4 Define the Boundaries of the Investigation

The objective of this step is to identify the sampling units (SUs) and to define the spatial and temporal elements of the investigation area. The boundaries of the investigation are delineated by combining the target population (population of interest) with the spatial and temporal boundaries. Implementing this step helps ensure the data are representative of the population. Spatial and temporal boundaries permit the identification of decision units (DUs), the smallest user-defined area(s) for which a decision will be made.

4.4.1 Spatial Boundaries

The spatial boundaries of the SCP RI sampling activities are as follows:

- Soil:
  - Lateral extent is the waste filled extent.
  - Vertical extent for surface soil is limited to 0-6 inches bgs.
  - Vertical extent for subsurface soil extends from the 6 inches bgs to 10 ft bgs.
- Reference Soil:
  - Lateral extent is the identified soil sampling reference area north of the Belt Parkway.
  - Vertical extent for surface soil is limited to 0-6 inches bgs.
  - Vertical extent for subsurface soil extends from the 6 inches bgs to 10 ft bgs.
- Groundwater:
  - Lateral extents are the two historic support areas where groundwater monitoring wells are planned.
  - Vertical extent is limited to the shallow unconfined aquifer.
- Sediment:
  - Lateral extent is the area encompassed by the existing on-Site surface water sampling locations (SW-01 through SW-05).
  - Vertical extent is the expected sampling interval from 0-6 inches below sediment surface (bss).

- Reference Sediment:
  - Lateral extent is the identified sediment sampling reference area north of the Belt Parkway.
  - Vertical extent is the expected sampling interval from 0-6 inches bss.
- Soil Gas and Landfill Gas:
  - Lateral extent for soil gas and landfill gas is a 200 ft wide buffer from the Site boundary adjacent to the residential areas.
  - Vertical extent for soil vapor is the vadose zone (4-5 ft bgs or adjusted shallower if groundwater is encountered shallower than 5 ft bgs).
- Radiological Anomalies:
  - Lateral extent is the waste filled extent.
  - Vertical extent for intrusive investigation of radiological anomalies is 5ft bgs.

The reference area for SCP is immediately to the north of the Site in an area that has not been developed or altered. Reference area sampling is being performed to understand the ambient levels of chemicals and radionuclides in environmental media outside of the Site to support conclusions regarding the attribution of chemicals and radionuclides detected in environmental media collected on Site (i.e., determining whether they are Site-Related or Not Site-Related). **Figure 4-1** provides a map of the Site and reference areas detailing the location of investigation activities to be performed during the SCP RI.

4.4.2 Temporal Boundaries

The temporal boundaries of the SCP RI sampling activities are as follows:

- Soil, Reference Soil, Sediment, Reference Sediment and Soil Gas
  - Concentrations of contaminants in soil, sediment, and soil gas are unlikely to vary seasonally; therefore, samples may be collected at any time during fieldwork.
- Groundwater
  - Only shallow groundwater will be investigated for the purpose of identifying if a release has occurred from operations in the historic support areas. Since shallow groundwater does not experience significant tidal influence, samples may be collected at any time during fieldwork.
- Radiological Anomalies
  - The current boundary of the waste filled extent at the Site, that corresponds to the area of potential disposition of radiological artifacts, is not expected to vary temporally; therefore, investigation activities may be performed at any time during fieldwork.
- Landfill Gas:

  - Concentrations of landfill gases vary seasonally with highest concentrations occurring in the warmer summer months (USEPA, 2005); therefore, samples should be collected during the warmer months of fieldwork.

SCP RI sampling activities will be performed as a part of a single event planned for Fall 2024. Fieldwork is expected to take approximately two months to complete.

4.4.3 Sampling Units

The SUs for SCP RI sampling activities are as follows:

- Soil
  - Surface Soil: Discrete surface soil samples (one at 0-6 inches bgs) will be collected from the approximate center location within the sampling unit of a 2-acre cell of a grid laid out across the waste fill extent (105 samples). The generation of the 2-acre grid and placement of planned sampling locations within grid cells was completed using Visual Sample Plan (VSP). Within each of the two historical support areas, one surface soil sampling location is bias placed in accordance with professional judgment in the location most likely to contain contamination based on knowledge of historic operations (2 samples).
  - Subsurface Soil: Up to two discrete subsurface soil samples will be collected from 10 ft bgs at each soil boring location. Soil borings will be advanced at the approximate center location within the sampling unit of a of a 2-acre cell of a grid laid out across the waste fill extent (105 samples). The generation of the 2-acre grid and placement of planned sampling locations within grid cells was completed using VSP. Within each of the two historical support areas, one soil boring location is bias placed in accordance with professional judgment in the location most likely to contain contamination based on knowledge of historic operations (2 samples). Discrete subsurface and discrete surface soil samples will be co-located.

- Reference Soil
  - Surface Soil: Discrete reference surface soil samples (one at 0-6 inches bgs) will be collected according to a systematic grid generated using VSP to place fifteen (15) samples within the identified soil reference area. Each discrete surface soil sample is considered a unique SU.
  - Subsurface Soil: Discrete reference subsurface soil samples will be collected at 10 ft bgs according to a systematic grid generated using VSP to place fifteen (15) samples within the identified soil reference area. Each discrete subsurface soil sample is considered a unique SU. Discrete subsurface and discrete surface soil samples will be co-located.
    Note: Reference soil will be analyzed for the same list of analytes as Site soil.
- Groundwater
  - Each new monitoring well in the historical support areas is considered a unique SU.

- Sediment

  - Each discrete sediment sampling location is considered a unique SU. A discrete sediment sample will be collected from each of the 2020 EE/CA on-Site surface water sampling locations (SW-01 through SW-05) at a depth of 0-6 inches bss.

- Reference Sediment
  - Each discrete reference sediment sampling location is considered a unique SU. Discrete sediment samples will be collected at intervals 200-250 ft apart along the stream in the sediment reference area north of the Belt Parkway (5 samples) at a depth of 0-6 inches bss. *Note: Reference sediment will be analyzed for the same list of analytes as Site sediment.*
- Soil Gas and Landfill Gas:
  - Each sampling location is considered a unique SU.
- Radiological Anomalies:
  - Waste filled extent with surveys being performed to an assumed depth of 1 ft bgs and intrusive investigations being advanced to a maximum depth of 5 ft bgs, as follows:
    - Full coverage GWS of all fire roads (plus a 10 ft buffer on either side) in the waste filled extent is considered a SU.
    - In-situ gamma spectroscopy performed in select areas, with each select area considered a unique SU.
    - Intrusive investigation locations for representative anomalies or anomaly areas with biased radiological measurements and soil samples collected at each location. Each representative anomaly of anomaly area is considered a unique SU.

4.4.4 Decision Units

The established DUs (see **Figure 2-14**) are as follows:

- Surface Soil
  - Area designated for future recreational use in the Gateway 2014 GMP/EIS
  - Area designated as future natural areas in the Gateway 2014 GMP/EIS
  - Reference Area
- Subsurface Soil
  - Area designated for future recreational use in the Gateway 2014 GMP/EIS
  - Area designated as future natural areas in the Gateway 2014 GMP/EIS
  - 1951 Support Area
  - 1954 Support Area
  - Reference Area

- Groundwater
  - Shallow groundwater within 1951 Support Area
  - Shallow groundwater within 1954 Support Area
- Sediment
  - Northern drainage ditch
  - Wetland near the Cove
  - Pool near northeastern corner of the Site
  - Reference Area
- Soil Gas
  - Site boundary adjacent to the residential area
- Landfill Gas
  - Site boundary adjacent to the residential area
- Radiological Anomalies
  - Area along Fire roads (plus a 10 ft buffer on either side) within the waste filled extent
  - Area designated for future recreational use in the Gateway 2014 GMP/EIS
  - Area designated as future natural areas in the Gateway 2014 GMP/EIS

4.5 Develop the Analytical Approach

The following types of samples will be collected and analyzed for the following parameters to address the principal investigation questions. Analytical methods are indicated in **Section 4.7**.

- Soil: Site and reference area surface and subsurface soil samples will be analyzed for VOCs, SVOCs, PAHs, metals (including mercury and hexavalent chromium), pesticides, PCBs, herbicides, dioxins and furans, and radionuclides (gross alpha/gross beta, uranium-238, radium-226, thorium-232 and isotopic uranium and thorium). 25% of surface soil samples will be analyzed for TOC.
- Soil investigated for radiological anomalies: Surface and subsurface soil samples will be analyzed for radionuclides (gross alpha/gross beta, uranium-238, radium-226, thorium-232 and isotopic uranium and thorium).
- Groundwater: Groundwater samples will be analyzed for VOCs, SVOCs, PAHs, metals (including mercury and hexavalent chromium), pesticides, PCBs, herbicides, dioxins and furans, and radionuclides (gross alpha/gross beta, total uranium, radium-226, and radium-228).
- Sediment: Site and reference area sediment samples will be analyzed for VOCs, SVOCs, PAHs, metals (including mercury and hexavalent chromium), pesticides, PCBs, herbicides, dioxins and furans, and radionuclides (gross alpha/gross beta, uranium-238, radium-226, thorium-232 and isotopic uranium and thorium). All sediment samples will be analyzed for TOC.

- Soil Gas: Soil gas samples will be analyzed for VOCs.

4.5.1 Decision or Estimation Parameters

Decision criteria for identifying COPCs and COPECs are discussed in Sections 4.2.2 and 4.2.3. Chemicals and radionuclides detected above the relevant decision criteria will be identified as COPCs and/or COPECs. **Section 8** provides details of the planned approach for completing the HHRA and SLERA. Both the HHRA and SLERA establish exposure point concentration (EPCs) as the 95% upper confidence limit (UCL) of the mean concentration for the relevant Site datasets. The EPCs will be combined with appropriate exposure parameters and toxicity values to calculate risk estimates to determine if concentrations could pose an unacceptable risk to human and/or ecological receptors. Also, EPCs and dose rates collected during focused investigations of radiological artifacts will be used in the dose assessment to estimate the potential radiological dose to human and ecological receptors.

4.5.2 Action Levels

Specific analytes, their respective screening benchmarks (i.e., MCLs, RSLs, and ESVs), and analytical detection limits are presented in **Tables 4-4 through 4-7**.

4.6 Specify Performance or Acceptance Criteria

The purpose of this step is to establish the criteria needed to maximize the ability of the investigation to obtain the data needed to answer the principal investigation question(s) accurately and with confidence.

4.6.1 Quality Assurance / Quality Control

QA/QC measures will be implemented during the investigation to minimize variability, mitigate the potential for false positive and/or false negative error, and increase accuracy and defensibility in the collected data. The analytical laboratory for SCP RI sampling is Eurofins, which has Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) and New York State certifications. QA/QC measures that will be implemented with respect to the analytical laboratory operations and field activities include:

- Field QC will be implemented and measured through collection of field QC samples, including field-designated matrix spike/matrix spike duplicates (MS/MSDs) (5% frequency), field duplicates (10% frequency), equipment blanks (10% frequency), and trip blanks (one per cooler with VOC samples); and implementation of sample collection, handling, and shipping standard operating procedures (SOPs) (presented in **Appendix B**).
- Environmental samples will be analyzed following laboratory SOPs (**Appendix C**) that employ appropriate QC checks to ensure precision and accuracy of data.
- Project-specific Data Quality Indicators (DQIs) —precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) — have been established to aid in assessing overall data quality. The DQIs specify the performance criteria, QC sample and/or activity that will be used to assess the performance criteria and the type of error (sampling,

analytical, or both) that will be used to assess data quality. The project-specific DQIs have been established for environmental media (soil, sediment, groundwater, and soil gas) to be sampled and laboratory analyses to be conducted. The project-specific DQIs for soil/sediment, groundwater, and soil gas are provided in **Tables 4-8, 4-9**, and **4-10** respectively.

Laboratory Quality Assurance / Quality Control and Samples

Laboratory QA/QC as well as laboratory QA/QC samples are outlined by the laboratory SOPs presented in **Appendix C**.

Field Quality Assurance / Quality Control

Field QC will be implemented and measured through the collection of field QC samples, including field-designated matrix spike/matrix spike duplicates (MS/MSDs) (5% frequency), field duplicates (10% frequency), equipment blanks (10% frequency), and trip blanks (one per cooler with VOC samples); and implementation of sample collection, equipment decontamination, handling, and shipping SOPs (presented in **Appendix B**). Field sampling personnel will properly identify samples collected in the field with an adhesive sample label attached to each sample container. The sample label will contain the Site name, field identification number, date, time, location of the sample collected, and identification of preservatives used. Sample information will be legibly printed with waterproof ink. The sample identification numbers will be used on field sheets, chain-of-custody forms, and other documentation records.

Field Quality Control Samples

Field QC samples will consist of field-designated MS/MSDs, field duplicates, and trip blanks. Field-designated MS/MSDs will be collected at the standard collection frequency of 5% (i.e., one MS/MSD per 20 samples). Field duplicates and equipment blanks will be collected at a frequency of 10%. Equipment blanks will only be performed for sampling conducted using non-dedicated sampling equipment. One trip blank will accompany each cooler shipped to the analytical laboratory that contains samples for VOC analysis. Samples will be collected in appropriate sample containers with appropriate preservatives and stored on ice at 4 degrees Celsius ($^{\circ}$C) ± 2 $^{\circ}$C. **Table 4-11** provides a summary of sample handling, including container requirements, number of containers, volume requirements, preservatives, and holding times.

Decontamination Procedures

Decontamination will be performed on equipment (e.g., drilling equipment, scoops, and pumps) that is to be reused at each sampling location. Decontamination will be performed before sampling at each sampling location to minimize the possibility for sample cross-contamination. Decontamination procedures are presented in **Appendix B**. Equipment blanks will be collected at a rate of 10% for sampling activities that use non-dedicated sampling equipment to demonstrate that equipment decontamination is being performed properly.

Instrument/Equipment Testing, Inspection, and Maintenance

Equipment that is to be used on Site will be inspected daily before use. Equipment inspection will follow the Equipment Inspection Worksheet (**Appendix D**). If the equipment is found to be deficient, the use of that equipment will be discontinued until proper maintenance can be performed and it passes a re-inspection. If the maintenance cannot be performed promptly or the equipment does not pass a re-inspection, the equipment will be replaced and the replacement will be inspected before use. Equipment testing, inspection, and maintenance guidelines are outlined in **Table 4-12**.

Instrument/Equipment Calibration and Frequency

Instruments to be used on Site will be, at a minimum, quality control checked daily, which may include daily calibration for specific instruments. Quality control checks may occur more frequently as the conditions require. Instrument calibration and frequency guidelines are outlined in **Table 4-12.**

Radiological measurement instrumentation will be inspected by the Site Radiation Safety Lead or designee to ensure its proper working condition prior to use. Field equipment and instruments will be properly protected against inclement weather conditions during the field investigation. At the end of each working day, field equipment and instruments will be removed from the field and placed in a dry location for overnight storage and charging, as appropriate to the instrument. An overview of the QC requirements for radiological field instrumentation is presented in **Table 4-12**.

Sodium iodide (NaI) detectors will be used to measure gamma radiation levels. SOPs will be used as the procedure for operation of these instruments. Radiation instrumentation used in the investigation will be maintained and calibrated to operate within manufacturer's specifications listed in SOPs to ensure the required sensitivity and precision. Specific calibrations and maintenance are conducted by a vendor authorized to perform the calibrations by an NRC or Agreement State licensed personnel trained on the equipment or by the manufacturer.

Operational procedures will be utilized for field instruments to verify the equipment is operating properly and used correctly in the field to produce accurate and reliable data. At a minimum, calibrations of radiation detection instruments will be performed annually and after repair. Field instrument checks will verify instrument response and will be performed at the beginning of each day, at a minimum. National Institute of Standards and Technology (NIST) traceable sources will be used for calibrations (i.e., for instruments that use a source). If the instrument checks reveal that the instrument is outside established accuracy limits (i.e., an acceptable range of +/- 20%), the instrument will be marked out of service. If necessary, the instrument will be returned to the manufacturer for immediate repair and servicing. At a minimum, calibration records will contain the following information:

- Instrument name and identification number (e.g., model and serial number);
- Manufacturer;
- Date of calibration;
- Calibration due date;
- Name of company and person performing the calibration;
- Calibration points;

- Results of the calibration; and
- Calibration source documentation (e.g., serial number, certification, and radionuclides).

Inspection/Acceptance of Supplies and Consumables

At the beginning and end of each day, the field personnel will inspect consumables to ascertain their condition and supply. If the field personnel determine that the condition or supply of the consumables will impact work performance, the Field Team Leader will procure additional consumables prior to the start of field activities the following day.

Special Training and Certification

Onsite field personnel will receive 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training (and associated 8-hour refresher training as necessary), Radiation Worker training in accordance with the Radiation Protection Plan (RPP), and additional training, if necessary, as outlined in the Site-Wide Accident Prevention Plan. These training requirements apply to field personnel that are working within the Site on a daily basis. Site visitors will receive project-specific awareness training prior to entering the Site and will be escorted at all times when on Site.

Data Quality Indicators Table

- DQIs for soil/sediment are presented in **Table 4-8**;
- DQIs for groundwater are presented in **Table 4-9**; and
- DQIs for soil gas are presented in **Table 4-10**.

4.6.2 Decision Error Limits and Uncertainty Evaluation

Uncertainty limits are proposed by establishing performance goals of the analytical data for precision, accuracy, repetitiveness, completeness, and comparability parameters. Uncertainty limits will be examined through statistical evaluation, which is an important tool used in the data assessment to determine:

- Whether the data meet the assumptions under which the DQO and the data collection design were developed.
- Whether the total error in the data is small enough to indicate that the data is of sufficient quality to allow the decision maker to use the data to support decisions within tolerable decision error rates.

Sample collection and measurement decision errors will be minimized by following the SOPs provided in **Appendices B and C** and documenting field activities that deviated from the SOPs. Similarly, laboratory analyses will follow the standard laboratory procedures and QA/QC samples will be collected to identify errors associated with sample collection and analyses. Ideally, laboratory limits would be less than the various risk-based screening levels that used to screen the analytical results. Analytical methods selected for this project optimize achievement of the required low reporting limits.

4.6.3 Data Validation and Usability

Data Verification

Data verification will be performed on 100% of the analytical data obtained during field activities. Data verification may be performed electronically or manually, or by a combination of both, and shall include, but is not limited to, the following:

- Sampling documentation (e.g., field records, chain-of-custody forms, etc.);
- Preservation summary and technical holding times;
- Presence of all analyses and analytes requested;
- Use of the required sample preparation and analysis procedures;
- Limit of quantitation (LOQ) and limit of detection (LOD) evaluated against the project requirements;
- The correctness of the concentration units; and
- Case narrative.

Data Validation

The data validation process builds on data verification. The laboratory case narrative, QC sample results, and calibrations will be reviewed and data qualifiers removed or added considering project knowledge for 100% of the data. Method-specific instrument calibration and QC parameters will be reviewed for compliance with calibration and for QC requirements.

The first data package for each analytical method and matrix will be specified for Stage IV data validation. The remaining data packages will be subject to Stage IIb data validation. Data Validation will be conducted in accordance with the processes summarized in **Table 4-13**. Validation qualifiers will be applied in accordance with National Functional Guidelines (NFG) for organic and inorganic data review. Validation of radionuclide data will be performed in accordance with USACE guidance (USACE, 2008). Methods for which no data validation guidelines exist will be validated following the NFG deemed most appropriate by the data validator.

An in-depth review of the raw data to verify accuracy will be performed on 10% of the data and include, but not limited to, the following:

- Instrument calibration and QC parameters (method-specific) (these will be reviewed for compliance with the criteria specified in the applicable Summary of Calibration and QC Procedures tables and flagged, as necessary);
- Review of raw data such as instrument print outs, preparation logs, and run logs;
- Review of system performance;
- Random check of calculations, including, but not limited to, sample and QC results, initial calibration response factors and relative standard deviations, calibration verification standard response factors, and percent differences or percent drifts from expected values;

- Random verification of sample results to the raw data;
- Check for interference problems or system performance problems;
- Estimated results (J-qualifiers); and
- Resolution by the laboratories of any identified problems, as necessary.

Assessment, which includes data validation, will be accomplished by the Project Chemist. The data assessment will identify out-of-control data points and omissions and coordinate with the laboratories to correct data deficiencies.

4.7 Develop the Plan for Obtaining the Data

Soil, sediment, groundwater, and soil gas samples will be collected and analyzed for a wide range of analytes; the planned analytical methods include:

- VOCs – SW 846 Method 8260; EPA Method TO-15
- SVOCs – SW 846 Method 8270D;
- PAHs – SW 846 Method 8270D-SIM;
- Metals (including mercury and hexavalent chromium) – SW 846 Method 6020; 7471; 7199/218.6
- Herbicides – SW 846 Method 8151A;
- Pesticides – SW 846 Method 8081B;
- PCBs – SW 846 Method 8082A;
- Dioxins/Furans – SW 846 Method 8290A;
- Uranium-238, Radium-226, and Thorium-232 – USEPA 901.1M;
- Isotopic Uranium and Thorium – HASL 300;
- Gross alpha and gross beta – USEPA 900.0;
- Total uranium for drinking water – USEPA 200.8; and
- Radium-226 and Radium-228 for drinking water – USEPA 903/904.

The FSP to collect soil, sediment, groundwater, and soil gas samples for laboratory analysis is presented in **Section 5**. Analytical data generated from this investigation will be provided from the analytical laboratories with electronic data deliverables. One hundred percent (100%) of the analytical data will be validated (See **Section 4.6.3**).

5 Field Sampling Plan

This FSP guides the data collection for the SCP RI. The planned investigation was developed using the DQO process included as **Section 4**. The following section describes investigation methods, sample media, sampling locations, sampling procedures, and analyses that will be performed. **Appendix B** presents the activity-specific SOPs that will supplement the sampling methods described in this section.

5.1 Soil Investigation Activities

This section describes the soil investigation activities that will be conducted to meet the objectives of the SCP RI. DPT will be used to advance 107 borings within the Site for the collection of continuous soil cores and soil samples. The planned DPT boring locations are presented on **Figure 4-1** and the coordinates for the planned 107 DPT boring locations are presented in **Table 5-1**. The soil investigation approach involves advancing the soil cores to a maximum depth of 10 ft bgs or the bottom of waste fill (whichever comes first). Additional reference area soil sampling will also be performed. Fifteen (15) surface and subsurface soil samples will be collected from the reference area north of the Belt Parkway (see **Figure 4-1)**. A Geoprobe® DT45 Dual-Tube sampling system will be used to collect continuous soil cores at each reference location in accordance with the Geoprobe® Dual-Tube Sampling System SOP (see **Appendix B**).

5.1.1 Geologic Logging and Field Screening of Soil Cores

Each soil core will be logged using the Unified Soil Classification System (USCS) to define geologic characteristics observed along the core. Each core will also be evaluated for the presence of waste fill. Based on the timeframe in which waste disposal operations occurred at SCP, the waste fill is nearly 60 years old and highly decomposed, which poses challenges in discerning soil horizons that contain waste fill. The identification of waste fill will be accomplished by observing pieces of debris that were less susceptible to decomposition, such as glass, masonry, ceramics, rubber, and metal, in the core.

As part of the soil boring advancement activities, the soil cores will be field screened for VOCs and methane gas using a MiniRAE 3000 photoionization detector (PID) and a flame ionization detector (FID), respectively; measurements will be collected along each soil core at a frequency of one reading every foot. The results of geologic logging and field screening of the soil cores will be recorded on boring logs, using the template presented in **Appendix D**.

5.1.2 Radiological Scanning of Open Soil Borings and Soil Cores

At each DPT soil boring location, a 2-inch outer diameter (O.D.) polyvinyl chloride (PVC) pipe (Schedule 40) will be placed into the borehole to prevent it from collapsing while a gamma scan is performed along the open borehole. Once the PVC tubing is secured in the open borehole, the vadose zone at each location will be surveyed for gamma radiation by performing a down-hole gamma logging (DGL) survey that will involve collecting gamma count rates at 1-ft intervals. The measurements will be collected using a 0.5-inch by 1-inch NaI gamma detector (Ludlum 44-62 or equivalent). The

measurements will be one-minute integrated counts reported in counts per minute (cpm). The data for each segment will be recorded on the boring logs using the template presented in **Appendix D**.

Radiological surveys will also be performed along the soil cores by performing a gamma scan of the soil core after it has been removed from the ground. Soil cores will be scanned in 1-ft increments using a three inch by three-inch NaI (Ludlum Model 44-20 or equivalent) connected to a Ludlum Model 2221 data logger (or equivalent). The measurements will be one-minute integrated counts reported in cpm at the highest location identified during the core scan for that interval. The data for each segment will be recorded on the boring logs using the template presented in **Appendix D**.

5.1.3 Soil Sampling

Surface and subsurface soil samples will be collected at each DPT boring location depicted in **Figure 4-1**. A surface soil sample will be collected from 0 to 6 inches bgs and analyzed for both chemical and radiological contamination. If waste fill is observed within the top two feet of the soil core, then two subsurface soil samples will be collected. The first subsurface soil sample will be collected from 0.5 ft to 2 ft bgs and the second subsurface soil core will be collected from 2 ft to 10 ft bgs within the vadose zone. If waste fill is not present within the top 2 ft of the soil core, then a single subsurface soil sample will be collected from the vadose zone between 0.5 ft to 10 ft bgs. Subsurface soil samples will be discrete, with each sample being collected over approximately 12 to 18 inches of the soil core. Subsurface radiological samples will be collected at the depth exhibiting the highest levels of gamma radiation, as determined by radiological scans of the soil core. In the unlikely event that the maximum count rate is duplicated at multiple locations along the core, professional judgment will be used to select the sampling interval and the resulting field decision will be noted on the boring log. Subsurface samples for chemical analysis will be biased to coincide with the subsurface interval that exhibits evidence of contamination within the soil core (e.g., based on visual observations, olfactory, elevated PID readings, etc.). If discernable contamination is not observed, the subsurface sample for chemical analysis will be collected from the mid-point of the sampling interval. Additional sampling will be performed to evaluate surface and subsurface soil in the reference area (see **Figure 4-1**). Following collection, samples will be immediately labeled and transferred to a cooler with ice and a trip blank, as required. Sampling equipment (spoon or scoop) will be stainless steel and will be decontaminated with Alconox/Liquinox cleaner and de-ionized water between each location. Resulting fluid will be containerized in a 55-gallon steel drum and disposed of as investigation derived waste (IDW).

Surface and subsurface DPT soil samples will be analyzed according to the analytical approach described in **Section 4.5**. Container type, quantities, required volumes, preservation requirements, and holding times for the soil samples are presented in **Table 4-11**. **Table 4-4** presents the screening benchmarks and laboratory limits for each soil analyte. **Tables 5-2 and 5-3** present the location ID, sample ID, field parameters to be measured, chemical analyses, and associated QC sample frequency for surface and subsurface soil samples that will be collected, respectively.

Soil cuttings will be handled as contamination, scanned for the presence of radiological artifacts (i.e., to segregate them from soil if present), containerized in 55-gallon steel drums, and transported to the IDW storage area. A temporary decontamination pad will be constructed in a centralized location of the Site with a berm and plastic sheeting. Drill tooling will be pressure washed and then decontaminated with

Alconox/Liquinox cleaner and water at the decontamination pad between the installation of each soil boring. Decontamination fluid will be containerized in a 55-gallon steel drum and disposed of as IDW.

5.2 Groundwater Investigation Activities

Permanent monitoring wells will be installed at 2 locations to investigate the historical support areas, (see **Figure 4-1**). The following section describes well installation and sampling activities planned to achieve the SCP RI sampling objectives for groundwater.

5.2.1 Monitoring Well Installation

Monitoring wells will be installed at the two locations depicted in **Figure 4-1**. The numbering scheme for monitoring wells accounts for the monitoring wells previously installed at SCP during the 2020 EE/CA, which have already been numbered through MW-21. The coordinates for each monitoring well are presented in **Table 5-1**. A shallow well will be installed at each location. Each shallow well will be constructed with a screen that spans the water level surface so that light non-aqueous phase liquid (LNAPL) can be detected if present.

Each groundwater monitoring well will be constructed with a 2-inch inner diameter (I.D.) schedule 40 PVC riser pipe using a DPT rig with 4.5-inch diameter DPT tooling. The shallow wells will be constructed with a 15-ft screened interval spanning the shallow water level surface. The longer screened interval was selected for the shallow wells to account for seasonal fluctuations in the groundwater surface. The well screen will include 0.010-inch (ten slot) slotted Schedule 40 PVC and the filter pack will be constructed using #0 silica sand and will be installed to approximately 2-ft above the screened interval. Each well will be sealed with a 2-ft layer of bentonite pellets which will be hydrated prior to grouting the well to the ground surface with Portland cement using the tremie method. The DPT rods will be extracted during the construction of the well to prevent the borehole from collapsing. Monitoring wells will be constructed as “flush mounts” (i.e., at/below grade well completions) with a secured well vault encased in concrete. Each well will be permanently labeled for identification and wells will be provided with locks.

Each monitoring well will be developed no sooner than 48 hours after installation. During development, water quality parameters will be observed and recorded every five (5) minutes. Well development will be performed until water quality parameters have stabilized (i.e., when three consecutive field parameter measurements of temperature, pH, specific conductivity, oxidation-reduction potential (ORP), salinity, and turbidity are within 10%) or until three well-volumes has been pumped from the well. Well development activities will be documented on the log sheets provided in **Appendix D**. Soil cuttings and development purge water will be containerized in 55-gallon steel drums, scanned for the presence of radiological contamination (i.e., soil will be scanned prior to being added to the drum and fluid drums will be scanned for external contamination), and transported to the IDW storage area. Drill tooling and well installation equipment will be pressure washed and then decontaminated with Alconox/Liquinox cleaner and water at the decontamination pad between the installation of each monitoring well. Decontamination fluid will be containerized in a 55-gallon steel drum and disposed of as IDW.

5.2.2 Groundwater Sampling

Groundwater samples will be collected no sooner than 24 hours after monitoring well development has been completed. Sampling will be performed using low-flow methods with a QED Sample Pro Bladder Pump #7944 and dedicated Teflon lined tubing. Static water level measurements will be collected prior to sampling each monitoring well. During purging and sampling, the submersible low-flow bladder pump will be placed at the mid-point of the submerged screen interval. During sampling, the purge rate will be adjusted as necessary to achieve flow between 0.1 to 0.5 liters per minute with minimal drawdown (i.e., a target of less than 0.1-meter).

Purge water will be directed through a flow-through cell housing a multi-parameter water quality meter (Horiba U-52). Parameters will be observed and recorded every five (5) minutes until water quality parameters have stabilized (i.e., when three consecutive field parameter measurements of temperature, pH, specific conductivity, dissolved oxygen, ORP, salinity, and turbidity are within 10%) or until the well has been pumped dry three (3) times. Well purging observations will be recorded on groundwater sampling log sheets which are provided in **Appendix D**. Groundwater samples will be unfiltered. Following sample collection, samples will be immediately labeled and transferred to a cooler with ice and a trip blank, as required. Plastic tubing will be disposed of between wells. Non-dedicated sampling equipment (i.e., the bladder pump) will be decontaminated with Alconox/Liquinox cleaner and deionized water using a triple-wash decontamination approach in between each well. Decontamination fluid and development/sampling purge water will be containerized in 55-gallon steel drums, which are scanned for the presence of external radiological contamination and transported to the IDW storage area.

Groundwater samples will be analyzed according to the analytical approach described in **Section 4.5**. Container type, quantities, required volumes preservation requirements, and holding time for the groundwater samples are presented in **Table 4-11**. **Table 4-5** presents the screening benchmarks, and laboratory limits for each individual groundwater analyte. **Table 5-4** presents the location identification (ID), sample ID, field parameters to be measured, chemical analyses, and associated QC sample frequency for each groundwater sample to be collected.

5.3 Sediment Investigation Activities

Sediment samples will be collected from five locations within the Site at the same locations as the five on-Site surface water samples collected during the 2020 EE/CA (SW-01 through SW-05). **Figure 4-1** shows the proposed sediment sampling locations and the coordinates for each are presented in **Table 5-1**. Additional reference area sediment sampling will be performed to evaluate sediment background. Five reference area sediment samples will be collected from a stream north of the Belt Parkway at intervals of 200-250 ft apart (see **Figure 4-1)**.

Sediment samples will be collected with either a stainless-steel slide hammer or a Ponar sediment grab sampler (depending on accessibility and sediment grain size at each sampling location). Sediment samples will be screened with a PID and a gamma radiation detector. Samples will be immediately labeled and transferred to a cooler with ice and a trip blank, as required. Sediment sampling activities will be documented using the log sheets provided in **Appendix D**. Non-dedicated sampling equipment will be decontaminated with Alconox/Liquinox cleaner and deionized water using a triple-wash decontamination approach in between each sampling location. Decontamination fluid will be containerized in 55-gallon

steel drums, which are scanned for the presence of external radiological contamination and transported to the IDW storage area.

Sediment samples will be analyzed according to the analytical approach described in Section 4.5. Container type, quantities, required volumes, preservation requirements, and technical holding times for the sediment samples are presented in **Table 4-11**. **Table 4-6** presents the screening benchmarks, and laboratory limits for each individual sediment analyte. **Table 5-5** presents the sample ID, field parameters, chemical analyses, and associated QC sample frequency for each sediment sample to be collected.

5.4 Soil Gas and Landfill Gas Investigation Activities

Ten (10) shallow soil gas probes will be installed using DPT to evaluate the Site boundary for VOCs and landfill gas. **Figure 4-1** shows the locations of planned soil gas probes and the coordinates for each are presented in **Table 5-1**. Each soil gas probe will be installed using DPT, with screens installed between 4 and 5 ft bgs. Each soil gas probe will consist of a 6-inch stainless-steel wire mesh screen connected to low density polyethylene (LDPE) tubing. At each soil gas probe location, field screening-level sampling/analyses will be conducted using a portable (direct reading) instrument (e.g., combustible gas indicator [GEM^{TM}2000 Plus with the targeted gas probes, or similar]) to determine the presence of CH_4, CO_2, O_2, CO, and H_2S. The GEM will also provide measurements of barometric pressure, temperature, and gas flow rate, at each soil gas probe. Soil gas samples will be collected using a VOC canister (summa canister) and sampler capable of filling an initially evacuated canister by action of the flow-controlled pump from vacuum to near atmospheric pressure. Both the summa canisters and samplers will be provided by the analytical laboratory. The filled canister will be properly labeled and packaged for shipment to the laboratory for VOC analysis via method TO-15.

Container type, quantities, required volumes, and technical holding times for soil gas samples are presented in **Table 4-11**. **Table 4-7** presents the screening benchmarks, and laboratory limits for VOCs in soil gas. **Table 5-6** presents the sample ID, chemical analyses, and associated QC sample frequency for each soil gas sample to be collected. **Appendix B** presents an SOP for the installation of soil gas probes and operation of the GEM for the evaluation of landfill gases.

5.5 Gamma Walkover Surveys

Gamma walkover surveys (GWSs) will be conducted along the fire roads (plus a 10 ft buffer on either side) (see **Figure 4-1**). GWS will be performed by qualified radiological control technicians using a 3-inch by 3-inch sodium iodide (NaI) detector connected to a Ludlum Model 2221 (or equivalent) and a global positioning system (GPS). The detector will be suspended approximately 4 inches over the surface being measured and will move at a rate of approximately 0.5 meters per second along paths approximately 1 meter apart. Following each day of surveying, the resulting data will be downloaded and exported to a text file that will include coordinates, instrument count rate, and log time for each point. Time-series plots will be generated and reviewed to ensure the survey instrumentation is operating properly. Geospatial modeling software will be used to develop count rate contours and a color-coded plot

using Surfer software. Geospatially correlated survey maps will be available to the project team on a near real-time basis. These plots will be reviewed daily to ensure sufficient survey coverage is maintained.

Personnel will designate a fixed GPS calibration point and will conduct daily quality control checks at the start of each workday. Internal checks will also be conducted to ensure proper communication between the Ludlum 2221 and the Trimble Geo 7X or equivalent to ensure that data is collected on a per second basis. Data will be extracted at the end of each day. The nearest governmental base station or base station with equivalent error will be used for differential correction before integration into the Site GIS.

The gamma count rate will be recorded every second during the GWSs along with the location of the measurement, the time of the measurement, and the elevation above mean sea level. Data will be evaluated using the Microsoft Excel® data analysis tool and ProUCL Version 5.1. The data analysis tool will be used to generate basic descriptive statistics. Summary tables will include mean, median, mode, standard deviation, standard variance, kurtosis, minimum, maximum, and sample population count. The mean and the standard deviation variables will be used in the calculation of the Z-score (described below). ProUCL software will be used to provide similar general statistics and generated percentile information for the data subsets. ProUCL will also be used to generate histograms, quantile-quantile plots, and scatter plots for each data subset.

Each data subset will be normalized by the calculation of Z-scores per the formula below

$$Z\text{-}score = \frac{\chi - \mu}{\sigma}$$

Where:

χ = result;
μ = mean of the population; and
σ = standard deviation.

For the data analysis, a Z-score greater than three standard deviations will be considered indicative of contamination. The review will combine observation of individual data points that exceed three standard deviations with any identifiable spatial patterns or trends that may indicate areas of relatively elevated activity.

5.6 Focused Investigations of Radiological Anomalies

Elevated locations identified during the GWSs will be pin-pointed and flagged to perform isotopic analysis of the localized areas of elevated radioactivity with an in-situ gamma spectroscopy system. A Trimble Geo 7X handheld device (or equivalent) will be used to reacquire the flagged location selected for focused investigation. A verification survey will be collected using the flagged location using a three-inch by three-inch NaI (Ludlum Model 44-20 or equivalent, shielded [if necessary]) connected to a Ludlum Model 2221 data logger (or equivalent); measurements will be logged for count rate and dose rate using a dose rate meter appropriate for the source fluency (e.g., Bicron Microrem detector or Ludlum Model 9 Ion Chamber) at waist level and ground surface. The AMETEK Ortec Trans-SPEC will be used for isotopic identification. The Trans-Spec is a high purity germanium gamma-ray spectrometer with a GEM Series P-type crystal that is 65 millimeter (mm) in diameter x 50 mm in length and >40% relative

efficiency. The resulting gamma spectrum will be analyzed using Ortec GammaVision for isotopic identification. GammaVision will analyze the data collected and provide the amount of radiation detected at each identified gamma energy. Since the gamma energy of a radionuclide is unique to the radionuclide, increased activity at a specific energy is an indication that a specific radionuclide is present. This information will be used to identify whether locations at the Site exhibit spectrum that may be attributed to the presence of a radiological anomaly (e.g., a discrete man-made artifact, monazite sand, a rock, or other natural feature) that requires additional intrusive investigation to evaluate the source of the radiation.

Following the collection of a gamma spectrum for radiological anomalies, focused investigations will be performed for representative anomalies or anomaly areas identified at the Site. Focused investigations will be prioritized based on the likelihood of radiological artifacts and/or distributed radiological contamination being present, which will be determined through evaluations of the GWS, dose rate measurements, and in-situ gamma spectroscopy results. Once locations have been selected, each focused investigation of radiological anomalies will proceed, as follows:

- Collect a surface soil sample for radiological analysis.
- Advance the investigation in 1 ft lifts through excavation (i.e., for suspected discrete radiological anomalies) or by collecting a 5 ft soil core using DPT (i.e., for suspected distributed radiological contamination). A shielded NaI connected to a Ludlum 2221 data logger (or equivalent) will be used to guide intrusive investigations. The shielded NaI detectors will be used to reduce radiation from other potential sources. The shield will be approximately 0.25 inches thick and will surround the lower 6 inches of the detector.
- Each investigation will be advanced to a maximum depth of 5 ft bgs.
- Remove the source of contamination or radiological artifact and collect a contact and 1 ft dose measurement of the item with a dose rate meter (i.e., to determine the activity of the artifact). A dose rate meter with sufficient capability will be used to determine an accurate dose rate of an item (e.g., Bicron Microrem, Bicron RSO-500, or equivalent).
- Collect a subsurface soil sample of the contaminated material or soil in direct contact with the radiological artifact for radiological analysis.
- Perform an assay of the artifact using the AMETEK Ortec Trans-SPEC detector (or equivalent) to generate a spectrum of gamma radiation. The resulting gamma spectrum will be analyzed using Ortec Gamma Vision for isotopic identification.
- Collect physical measurements of the artifact (size/weight) and photolog the radiological artifact or source of contamination.

The data resulting from these focused investigations include analytical results for radionuclides via laboratory analysis (i.e., methods 900, 901.1M, and alpha spectroscopy [HASL 300]) , count rates, and dose rates (waist level and contact) for both surface and subsurface soil at each anomaly location. In addition, physical measurements, a photolog, and a gamma spectrum will be generated for each recovered radiological artifact or source of contamination. The focused investigation results will support the evaluation of the nature and extent of contamination as well as the dose assessment, which will be

included in the risk assessment. **Appendix B** includes SOPs for the various activities that will be performed as part of these focused investigations. Radiological artifacts or other radiological material resulting from these investigations will be managed in accordance with the project RPP.

5.7 Investigation Derived Waste Handling

IDW generated during Site field activities will be managed pursuant to applicable Federal, State, and local regulations. Solid IDW will include soil from soil borings and monitoring well installations and personal protective equipment (PPE) that is contaminated. Liquid IDW will include monitoring well development water, purge water from sampling, and decontamination fluids generated at the decontamination pad. Solid and liquid IDW will be stored in 55-gallon drums meeting Department of Transportation requirements and staged in the designated IDW storage location. A composite solid and liquid sample will be collected from each IDW drum to support a waste profile for subsequent offsite disposal.

All IDW drums will be transported, staged, and inventoried within a secured, fenced area established at the Site. Additional details relating to the management of IDW are included in **Appendix B**.

5.8 Handling of Radiological Contamination

Recovered radiological artifacts will be stored in shielded containers and radiological soil contamination will be stored in 55-gallon drums. The drums and containers will be secured onsite in a locked conex box (or similar locking structure) to prevent tampering, pending sampling and disposition. If the radiation dose at any exterior wall of the conex box (or similar locking structure) is 2 mrem/hr or greater and cannot be reduced by use of shielding or placement of containers, the conex box will be posted accordingly per radiological requirements associated with Tidewater's NY State Radiological Materials License.

5.9 Health and Safety

Field sampling activities described in **Section 5** will be performed in accordance with the Final Gateway-Wide Accident Prevention Plan (G2S, 2022).

5.10 Sample Handling

This section describes the sample handling protocol for environmental samples collected during the SCP RI.

5.10.1 Sample Designation

Each sample will receive a unique designator. (i.e., the field sample ID). Unique designators will be an alpha-numeric combination that signifies the sample location or decision area, matrix, and/or depth. Sample handling and designation will conform to the procedure presented in **Appendix B**.

5.10.2 Sample Labeling

Field sampling personnel will properly identify samples collected in the field with an adhesive sample label attached to each sample container. The sample label will contain the Site/project name, sample designation (field sample ID), date, time, sample location, sampler's initials, analyses required, and identification of preservatives used. Sample information will be legibly printed with waterproof ink. The sample designation will be used on field sheets, chain-of-custody forms, and other documentation.

A sample numbering system will be used to uniquely identify each sample collected and submitted for analysis. The purpose of the numbering system is to assist in the tracking of samples and facilitate retrieval of analytical results. Sample identification numbers will be used on sample labels, chain-of-custody forms, field logbooks, and other applicable documentation. All sample identification numbers will be recorded in the field logbook along with the depth of samples if collected in the subsurface. Sample labeling for field samples (including duplicates) and water quality control samples follows.

Field and Duplicate Samples. The general labeling scheme for normal field samples will be as follows:

- Soil Samples: XXX-TTZZZ-D
- Soil Samples focused radiological investigation: XXX-TTYYYY-#-D
- Groundwater Samples: XXX-TTZZZ
- Sediment Samples: XXX-TTZZZ

where:

1. XXX- = **"SCP-"** to be used for samples within the SCP Site. "**REF-**" to be used for reference samples.
2. TTZZZ = Location ID (5 characters) – unique identifier for the sampling locations.

   2a. TT = Sample Location Type: **SE** – sediment location; **SS**– surface soil sample; **SU**– sub-surface soil sample; **SG** – soil gas sample; and **MW** – permanent monitoring well;

   2b. ZZZ = A sequential three-digit integer starting with 001 and running through 999. Unique ID assigned by G2S to identify the sampling location.

   - Surface and subsurface soil samples collected at the same location (i.e., soil boring) will be assigned the same location ID.
   - Monitoring well numbering will continue the sequential numbering established by the 2020 EE/CA.

   2c. D = Integer identifying approximate depth of subsurface soil sample in ft bgs. For surface soil samples, the D suffix is omitted.

3. TTYYYY = Location ID (6 characters) - unique identifier for the sampling locations.

   3a. TT = Sample Location Type: **SS**– surface soil sample; and **SU**– sub-surface soil sample.

   3b. YYYY = Alpha-numeric grid identification. The 1-acre alpha numeric-grid will be established following the gamma walkover surveys.

3c. # = Intrusive investigation number

3d. D = Integer identifying approximate depth of subsurface soil sample in ft bgs. For surface soil samples, the D suffix is omitted.

Field duplicate sample IDs will include "DUP" at the end of the associated field or parent ID. In addition to field duplicates, MS/MSDs will be collected at a rate of 5%. The MS/MSD samples will be identified on the chain-of-custody forms.

Examples of field sample IDs for surface and subsurface soil samples are as follows:

- For Soil Boring Location 003: **SCP-SS003** (surface soil sample); **SCP-SS003-DUP** (surface soil sample duplicate); **SCP-SU003-3** (subsurface soil sample collected at 3-ft bgs); **SCP-SU003-3-DUP** (sub-surface soil sample duplicate collected at 3-ft bgs).

Examples of field sample IDs for sediment samples collected are as follows:

- For Sediment Location 002: **SCP- SE002** (sediment sample); and **SCP-SE002-DUP** (sediment sample duplicate)

Examples of field sample IDs for groundwater samples are as follows:

- For groundwater sample collected at Monitoring Well MW022: **SCP-MW022**.

Water Quality Control Samples. The labeling scheme for trip and equipment blanks is as follows:

- Field Sample ID = **XXXTTMMDDYY-CCC**

where:

1. XXX = SCP to be used for all samples.
2. TT = QC Sample Type. **TB** – trip blank; **EB** – equipment blank.
3. MMDDYY = Sample Date.
4. CCC = Sample Counter. A sequential two-digit integer starting with 001. Unique ID assigned by G2S to be used if multiple samples of a particular type (trip blank) are collected on the same day.

Examples of field sample IDs for water quality control samples are as follows:

- For trip blank #1 collected on May 7, 2024: **SCPTB050724-001**.
- For trip blank #2 collected on May 7, 2024: **SCPTB050724-002**.
- For equipment blank #1 collected on June 20, 2024: **SCPEB062024-001**.
- For equipment blank #2 collected on June 20, 2024: **SCPEB062024-002.**

6 Data Management

This section summarizes the data management procedures that will be implemented for SCP RI sampling, specifically:

- Field documentation procedures;
- The analytical laboratories' electronic data deliverables (EDDs);
- Project file management and retention; and
- The project database.

6.1 Field Documentation Procedures

Fieldwork will include fully documented sample collection, preservation, and handling procedures as defined in the FSP (**Section 5**). A number of different documents will be completed for the fieldwork. The documents will provide a summary of the sample collection procedures and conditions, shipment method, analyses requested, sample custody history, and field technique improvements as required. Field documentation will include:

- Field logbooks;
- Sample collection forms;
- chain-of-custody; and
- Field QC reports.

Direct read data and/or measurements collected during fieldwork will be written into the field logbook or on customized and numbered field forms, immediately upon collection of measurements. Notations will be written in indelible ink and entries will be signed and dated. If entries must be changed, the reason for the change shall be noted and the change should not obscure the original entry (e.g., a single line drawn through text or an X through figures, tables, or maps). The change will be initialed and dated by the responsible person. If space is available, revisions will be added to the same page. Otherwise, the page where the revision is entered will be noted. Any lost, damaged, or voided field logbooks will be reported to the PM immediately.

Field records will be collected and verified daily by the Site Supervisor (or designee), who will review the data for completeness, accuracy, legibility, and comparability with other data collected, and verify the field data records have been signed and dated. Based on this review, the Site Supervisor will direct field staff to make necessary corrections to the record and to initial and date the corrections. Any omissions or inconsistencies discovered will be resolved by the Site Supervisor, who will seek clarification from the field personnel responsible for data collection.

After data reduction and entry into the project database, field data will be verified by qualified personnel for completeness, consistency with hardcopy records, and anomalous values. Field data, including both electronic and hardcopy documentation, will be reviewed by the Site Supervisor and/or PM prior to

inclusion in technical reports, and may be reviewed by the Project Chemist (or technical designee) as part of ongoing QC review of project activities.

6.2 Analytical Laboratory Electronic Data Deliverables

Analytical data from the laboratories for the SCP RI will be managed using ADR.Net and EDMSi. The ADR.Net software will be used to perform data verification, electronic data deliverable (EDD) compliance checks and automated review/validation and EDMSi will be used as the project database. Analytical data will be delivered by the laboratory in the ADR EDD format and verified for content and format compliance prior to delivery using the ADR.Net software. The ADR EDD format specifications and ADR.Net software are available from Laboratory Data Consultants, Inc (LDC) (http://www.lab-data.com/). The LDC Data Manager will provide Eurofins with the ADR.Net Project eQAPP containing the analytical requirements, data quality indicators and required valid values before the first sampling event begins.

EDD files received from the laboratory will be stored in the project file archive (see **Section 6.3**). The LDC Data Manager will verify the EDD is error free using the ADR.Net software. If error free, the EDD will be validated and loaded into the project EDMSi database. The laboratory will be contacted for corrective action if errors are found in the EDD. The laboratory will also be required to provide .pdf document files of the laboratory final data reports. The laboratory will be required to make available any supplemental information (e.g., chromatograms, instrument calibrations) upon request. Electronic data submitted by the laboratory will be required to be error-free and in complete agreement with the hardcopy data. Data files will be delivered via e-mail, or by posting on LDC Advantage, a secure project portal made available by Laboratory Data Consultants, Inc. The data files will be submitted with a transmittal letter from each laboratory that certifies that the files agree with hardcopy data reports and have been found to be free of errors using the ADR.Net software and ADR EDD format referenced above. The laboratory, at their cost, will be required to correct any errors identified.

6.3 Project File Management and Repository

Electronic Data. Electronic files provided by the analytical laboratories and GIS data layers and historical records/documents (including previous study reports, historical drawings and maps, and related items) provided by USACE or other stakeholders will be securely stored on a secure private network located at the Tidewater office (i.e., G2S JV member) in Elkridge, Maryland. Access to these files is restricted to only those personnel with key responsibilities to the project and who have been granted authority by the G2S PM.

Hardcopy Data. Various hardcopy files, including the Task Order and any modifications, correspondence, including meeting minutes and monthly reports, relevant records, reports, logs, field logbooks, photographs, subcontractor reports, data reviews, draft submittals, responses to comments and final submittals, and correspondence will also be stored within the secure G2S project files. The project files are located at the Tidewater office (i.e., G2S JV member) in Elkridge, Maryland. Access to these offices is limited to G2S personnel though a door security system and employees and visitors are badged.

The project information files will include, at a minimum:

- Field logbooks;
- Field data and data deliverables;
- Photographs;
- Drawings;
- Laboratory data deliverables;
- Data validation reports;
- Data quality assessment reports;
- Progress reports, QA reports, interim project reports; and
- Custody documentation (e.g., tags, forms, air bills).

In addition to the above information, the project files will also maintain contractual information. This includes, but is not limited to, contract information, cost proposal information, and invoice records.

6.4 Project Database

Field sampling and laboratory data will be compiled in an EDMSi (http://www.lab-data.com/) database. EDMSi is an industry-standard environmental data management platform, which will allow the project team to rapidly integrate, verify, and report on sampling and lab test results. EDMSi is cloud-based using SQL storage on elastic servers along with the latest encryption technology. Therefore, G2S is always using the latest version including features and security. Access to the EDMSi database will be controlled so that only the LDC Data Manager and other trained personnel designated by G2S will be allowed to view, add, edit, remove, or export data. The EDMSi database will be available to select project team staff for reporting purposes, and the LDC Data Manager will verify that these staff are trained to perform these tasks accurately.

7 Assessment and Oversight

This section describes the measures that will be employed to ensure that this SAP is implemented properly. These include conducting planned and documented performance audits for field operations to assess the accuracy of the measurement systems, to determine the effectiveness of QA/QC procedures outlined in **Section 4.6,** and compliance with project SOPs.

7.1 Assessment and Corrective Actions

The assessment and corrective actions will be implemented as follows:

- The PM will have the ultimate responsibility for implementing project QA/QC procedures. USACE will provide QA oversight and will review the work being conducted.
- USACE QA personnel will meet with the field supervisor and/or field staff to assess adherence with SAP requirements, review field forms related to the sampling that was performed, and review of the chain of custody (COC) forms submitted with samples.
- During fieldwork, USACE will communicate deficiencies in a timely manner. G2S will investigate any deficiency noted, determine the cause of the condition identified in the finding, schedule corrective action (including measures to prevent recurrence), evaluate the impact of the finding on completed work, and notify USACE of action taken or planned. The PM or designee will be responsible for verifying and documenting completion of the corrective action.

7.1.1 Field Audit and Response Actions

USACE will review the records of field operations to verify that field-related activities are performed in accordance with appropriate project procedures. Items to be examined will, as appropriate, include:

- Availability and implementation of approved SOPs;
- Calibration and operation of equipment;
- Labeling, packaging, storage, and shipping of samples;
- Performance documentation and checking;
- Field documentation and the collection of field quality control samples, including duplicates/replicates and field quality control samples; and

USACE will document noted deviations from the SAP, which will be communicated to the PM. The PM will be responsible for identifying necessary corrective actions, communicating the corrective actions to the field team, and verifying the corrective actions have been implemented.

7.2 Quality Assessment Reporting

In addition to the performance audit reporting discussed in **Section 7.1**, QA reports will be generated for activities discussed in the following sections.

7.2.1 Data Verification

Data verification procedures described in **Section 4.6.3** will be conducted on each sample data group (SDG) by the Project Chemist as sample data packages are generated by the laboratories. Verification activity findings will be included along with data summaries in the RI Report. Issues potentially affecting the usability of data will be reported by the Project Chemist to the PM as soon as they are identified so that appropriate corrective actions can be identified and implemented.

7.2.2 Data Validation

Data validation procedures described in **Section 4.6.3** will be conducted on each SDG by the Project Chemist as sample data packages are generated by the laboratories. Validation activity findings will be included along with data summaries in the RI Report. Issues potentially affecting the usability of data will be reported by the Project Chemist to the PM as soon as they are identified so that appropriate corrective actions can be identified and implemented.

7.3 Reconciliation with DQOs and Data Usability

An assessment of data quality will be conducted to determine whether the project DQOs have been achieved. The Project Chemist will document the results of the assessment, including whether any changes are necessary to the DQOs because data do not meet usability criteria, in data quality assessment reports that will be included in the RI Report.

The data usability assessment will include a review of the sampling and analysis activities in comparison to the project DQOs. The results of the data validation will be reviewed to identify specific limitations to the data (i.e., results qualified as estimated [J/UJ] or rejected [R]) for particular uses, such as data interpretation, analysis, and/or risk assessment. The assessment will also consist of a comprehensive evaluation of how the data meet the DQIs (PARCCS) to identify potential or actual problems with the data collection process and impacts to data so that corrective measure can be identified and implemented. Data that do not satisfy DQIs will be evaluated to determine the potential impacts based on the extent of the deficiencies and the importance of the data in the overall context of the investigation. Corrective measures will be implemented including conducting additional field measurements and resampling, as warranted, to ensure that data are valid, legally defensible, of known quality, and can be used without limitations as qualified to meet the investigation DQOs.

8 Investigation Outputs

After completion of RI field activities at SCP, results of the 2020 EE/CA investigation (JCO, 2020) will be combined with the additional data collection efforts described in this SAP to support the development of the RI Report. The RI Report will be developed in accordance with USEPA Guidance (USEPA, 1988) and will present a detailed summary of the nature and extent of contamination, an evaluation of contaminant fate and transport, and risk assessments.

A baseline HHRA and SLERA will be performed as components of the RI. The HHRA will evaluate potential human receptors and exposure pathways, as identified in the pathway-receptor diagram presented in **Figure 2-16** and quantify noncancer and cancer risks that may result from exposure to the COPCs in Site media. The HHRA will be conducted in accordance with:

- USEPA Superfund risk assessment guidelines presented in EPA/540/1-89-002, Risk Assessment Guidance for Superfund, Volume 1, Human Health Evaluation Manual (Part A) (Interim Final) (USEPA, 1989);
- and other USEPA HHRA risk assessment guidance.

The SLERA will follow NPS and USEPA ecological risk assessment methodology, including:

- USEPA Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments, EPA 540-R-97-006 (USEPA, 1997);
- USEPA ECO Update: The Role of Screening-Level Risk Assessments and Refining Contaminants of Concern in Baseline Ecological Risk Assessments, EPA 540/F-01/014 (USEPA, 2001b);
- NPS Protocol for the Selection and Use of Ecological Screening Values for Non-Radiological Analytes. Revision 3 (NPS, 2018c);
- NPS Protocol for the Selection and Use of Ecological Screening Values for Radionuclides. Revision. 1 (NPS, 2018d);
- and other pertinent ecological risk assessment guidelines.

This evaluation may also include a refinement step, in which the constituents retained by the conservative approach of the SLERA are re-evaluated using a broader array of exposure, effects, and contaminant distribution and concentration data to define potential risks more accurately.

The RI Report will summarize the results of the nature and extent evaluation, fate and transport evaluation, and the risk assessments to support conclusions and a recommended path forward for the Site under CERCLA.

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Figures

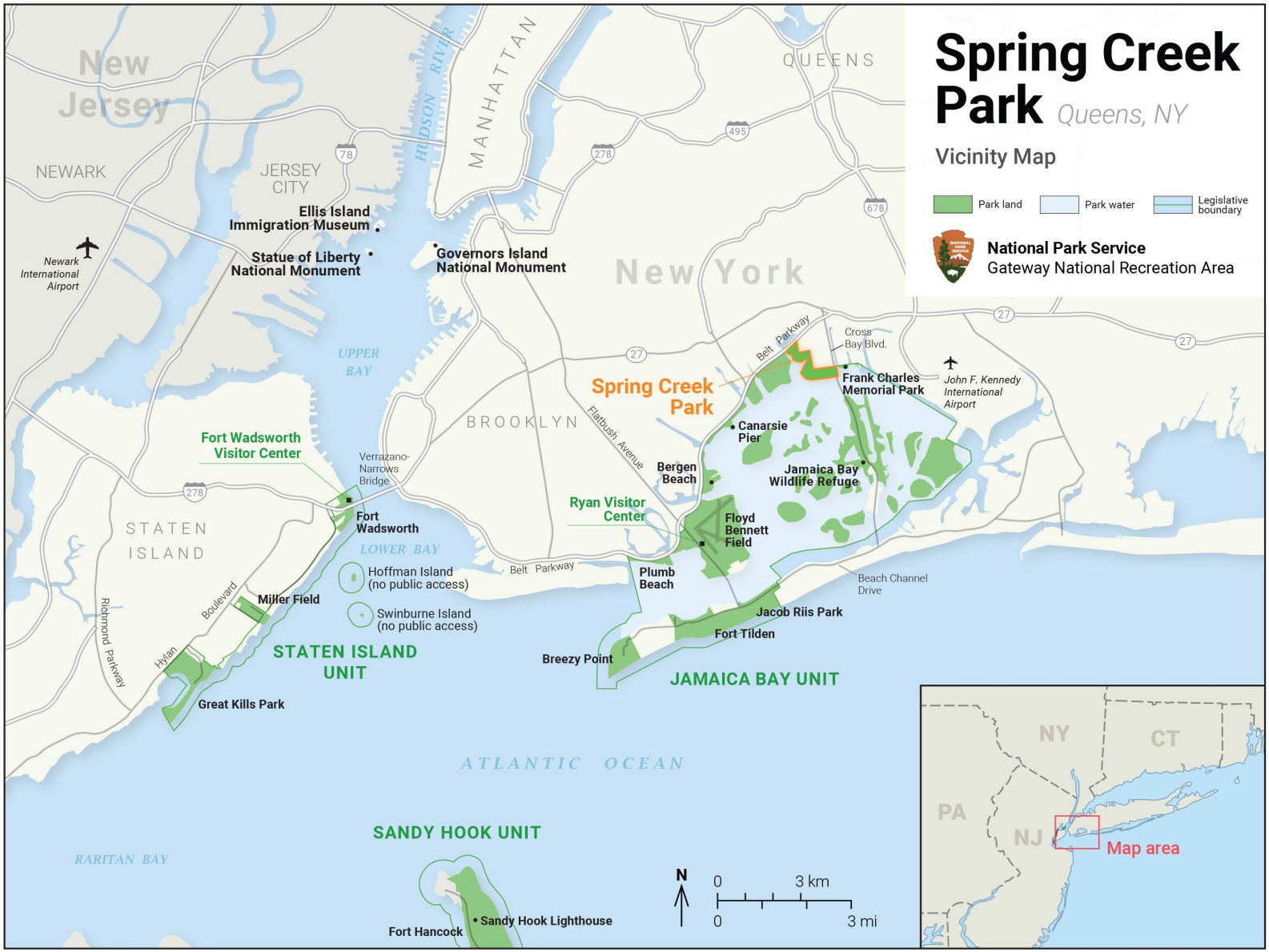
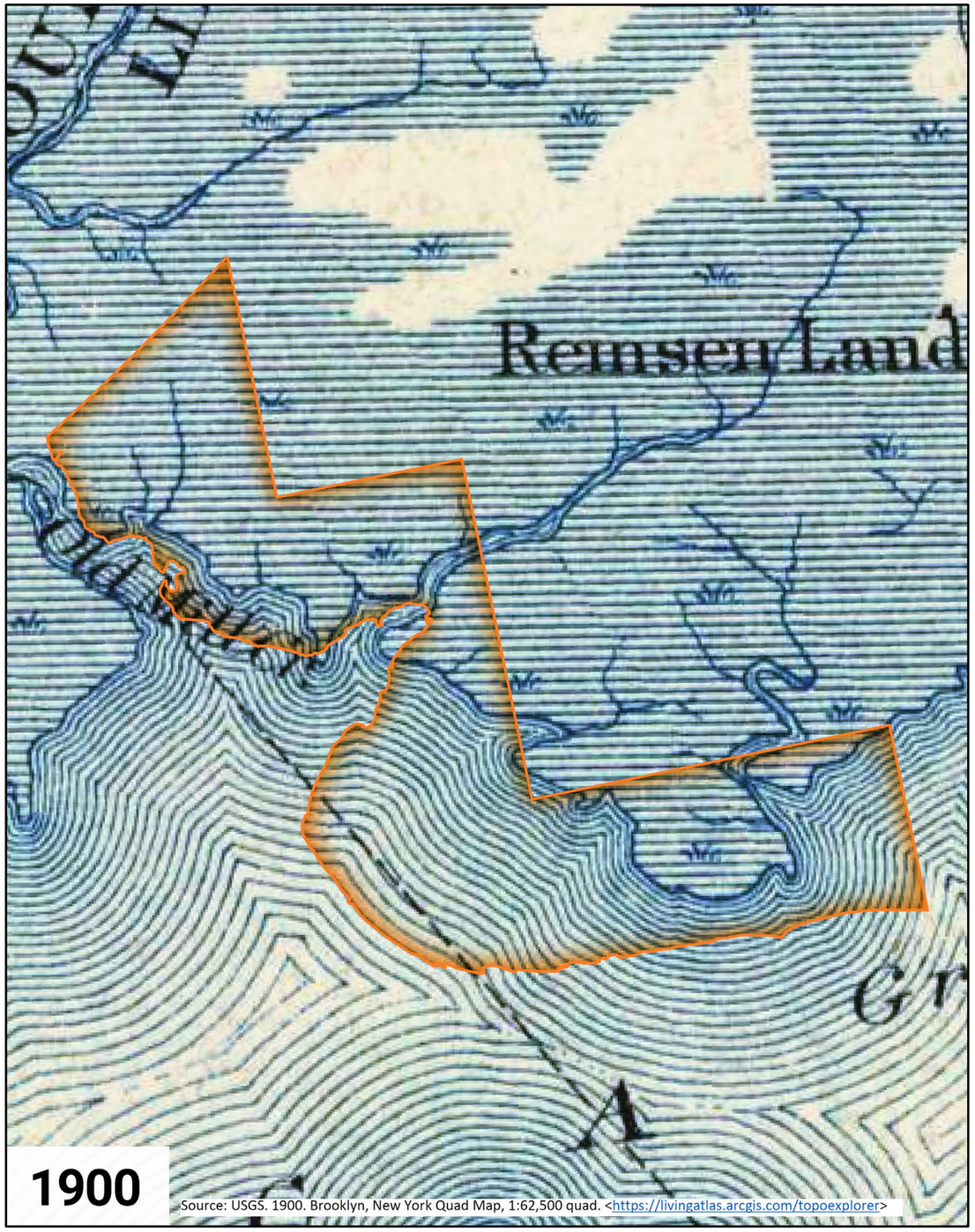


Figure 1-1. Vicinity Map Showing Gateway National Recreation Area and Spring Creek Park

Figure 1-2. Map of the Spring Creek Park Site



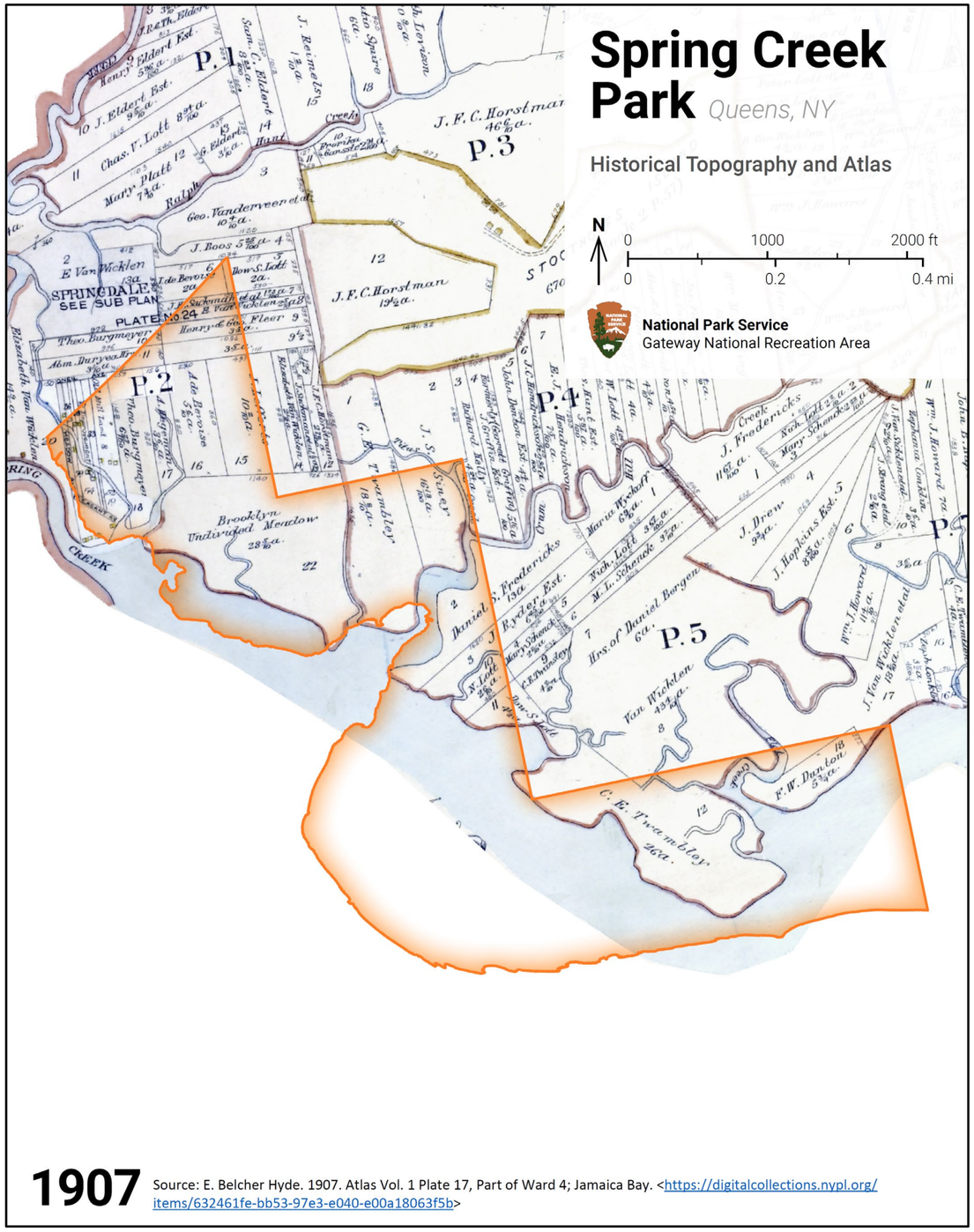
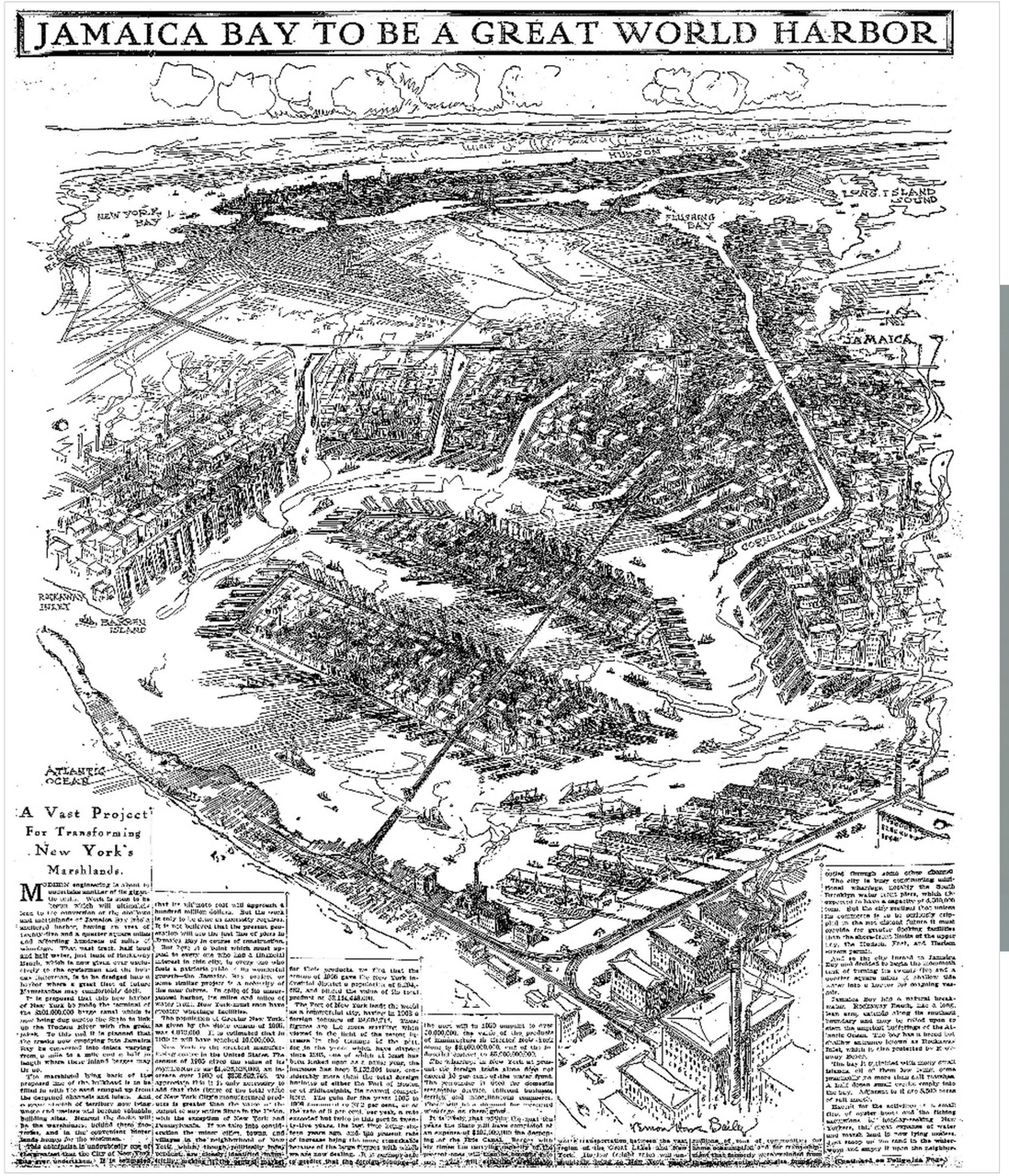
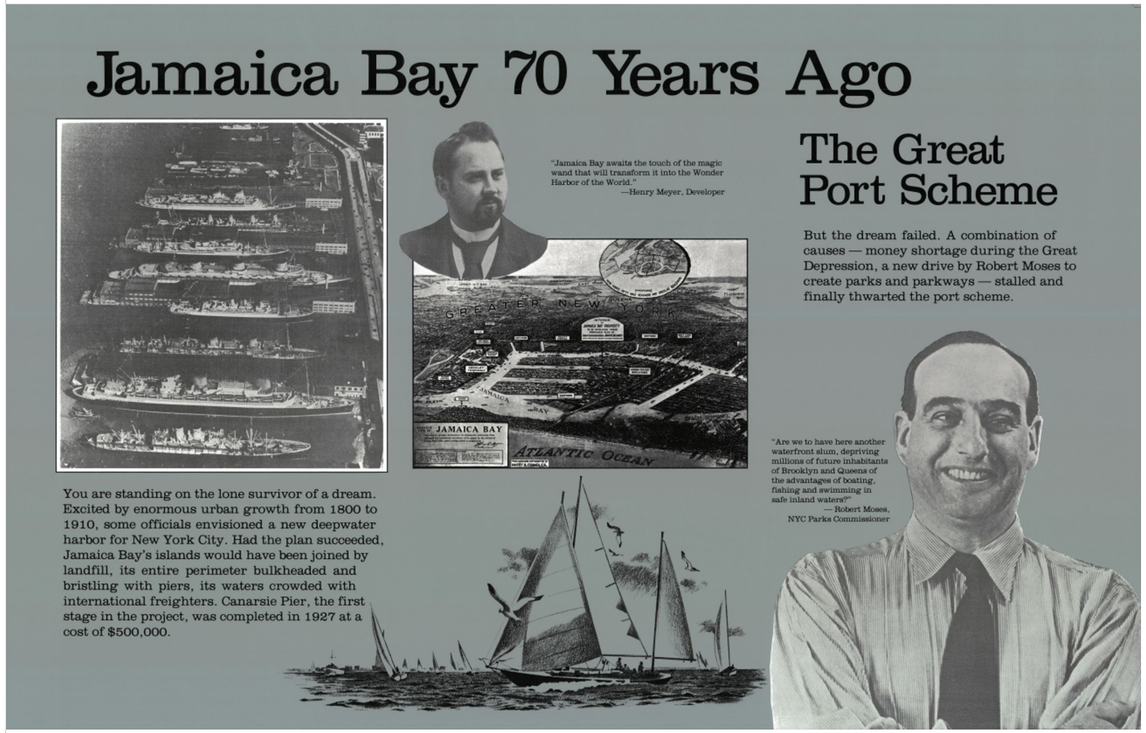


Figure 2-1. 1900 Topography Map and 1907 Atlas of Spring Creek Park


JAMAICA BAY TO BE A GREAT WORLD HARBOR

A Vast Project For Transforming New York's Marshlands.

Jamaica Bay Improvement Plan (NYT, 1910)


Jamaica Bay 70 Years Ago

"Jamaica Bay awaits the touch of the magic wand that will transform it into the Wonder Harbor of the World."
—Henry Meyer, Developer

The Great Port Scheme

But the dream failed. A combination of causes — money shortage during the Great Depression, a new drive by Robert Moses to create parks and parkways — stalled and finally thwarted the port scheme.

"Are we to have here another waterfront slum, depriving millions of future inhabitants of Brooklyn and Queens of the advantages of boating, fishing and swimming in safe inland waters?"
— Robert Moses, NYC Parks Commissioner

You are standing on the lone survivor of a dream. Excited by enormous urban growth from 1800 to 1910, some officials envisioned a new deepwater harbor for New York City. Had the plan succeeded, Jamaica Bay's islands would have been joined by landfill, its entire perimeter bulkheaded and bristling with piers, its waters crowded with international freighters. Canarsie Pier, the first stage in the project, was completed in 1927 at a cost of $500,000.

Great Port Scheme (NPS, 1990)

Figure 2-2. Historical Plans for Jamaica Bay

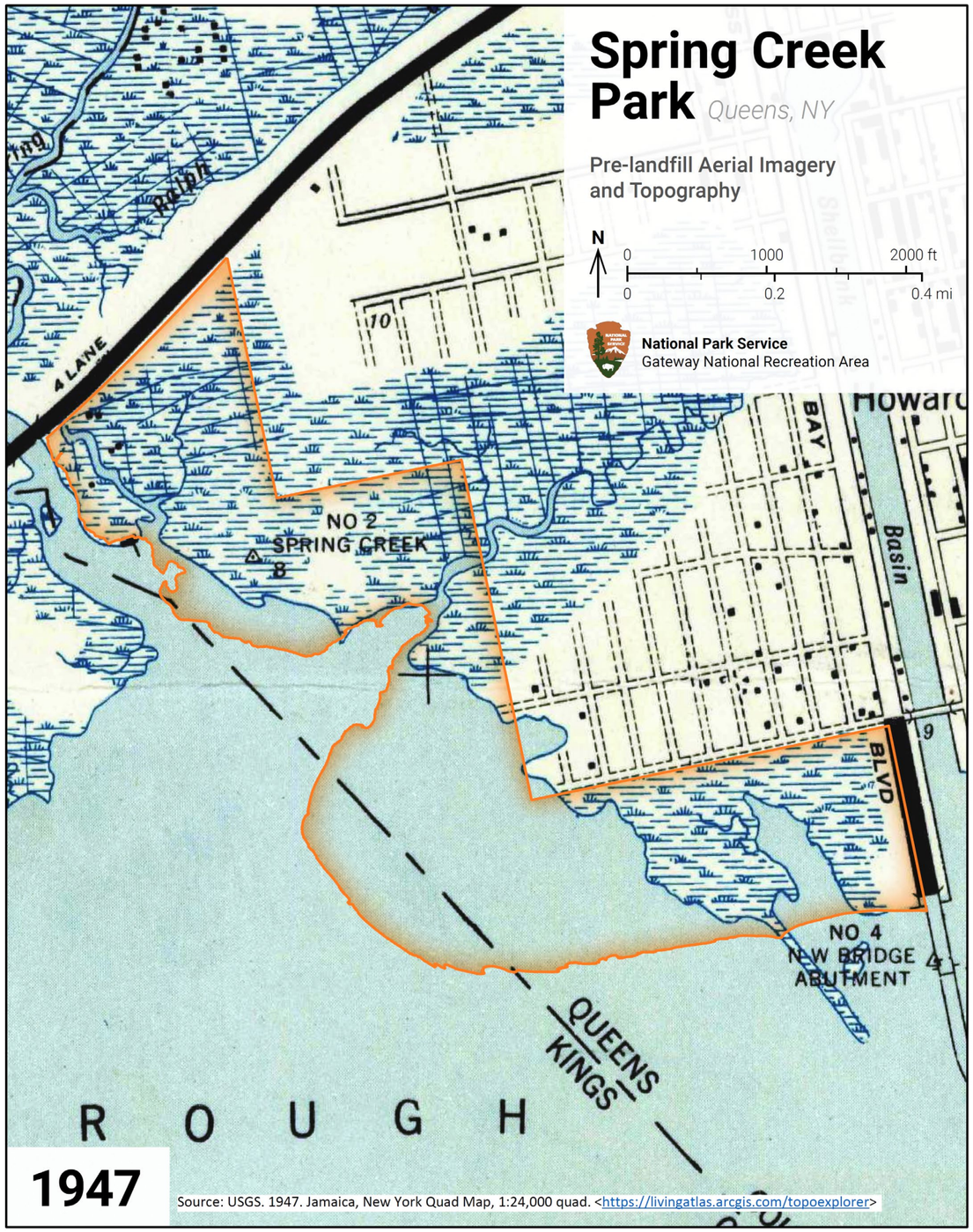


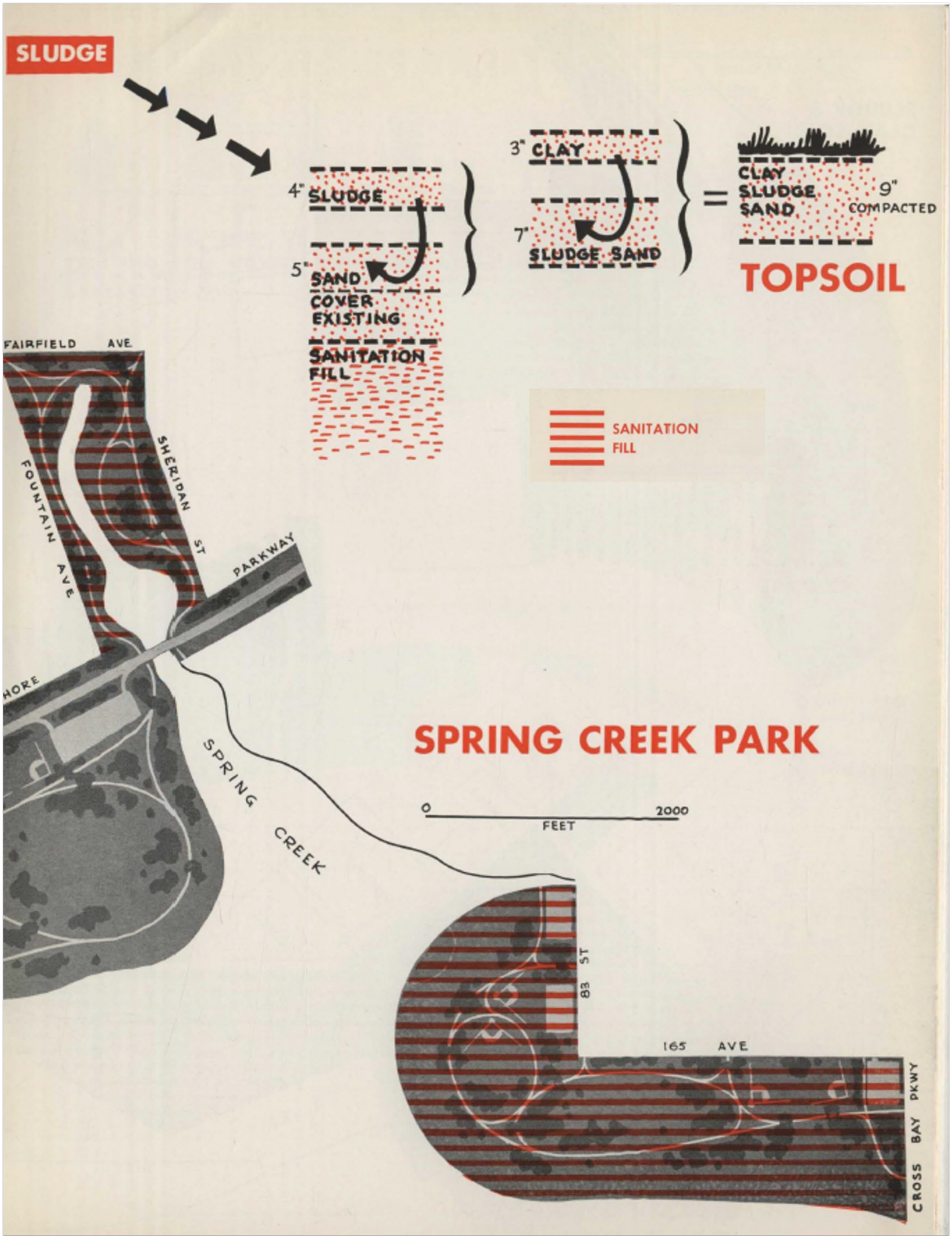
Figure 2-3. 1924 Aerial Imagery and 1947 Topography Map of Spring Creek Park

Figure 2-4. 1951 Aerial Imagery and 1954 Aerial Imagery of Spring Creek Park

Dump truck enters landfill area at Spring Creek, on Jamaica Bay. (Tress, 1973a)

Single auto axel cast off in landfill operation at Spring Creek, on Jamaica Bay. (Tress, 1973b)

Figure 2-5. Photos of Landfilling Operations at Spring Creek Park



Memo to Mayor Impellitteri: Reclamation; topsoil, cover [and] fill. (NYCDP, 1950)

Figure 2-6. 1950 Illustrated Map of Spring Creek Park and Schematic of Soil Amendment Process

Figure 2-7. 1961 Aerial Imagery and 1966 Aerial Imagery of Spring Creek Park

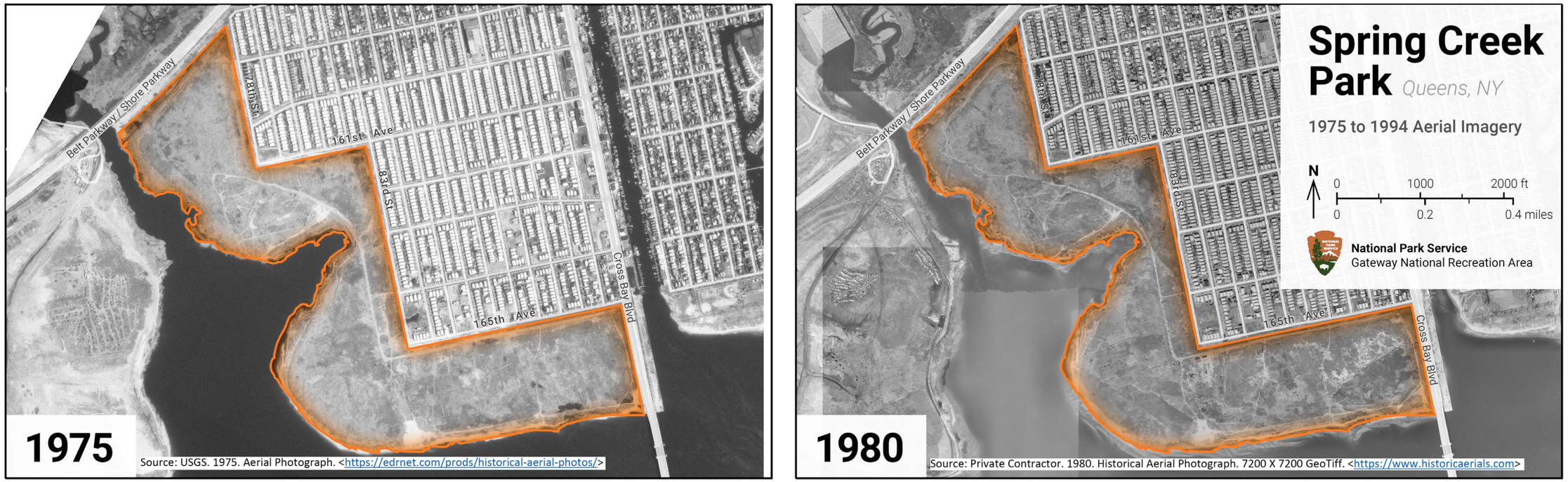
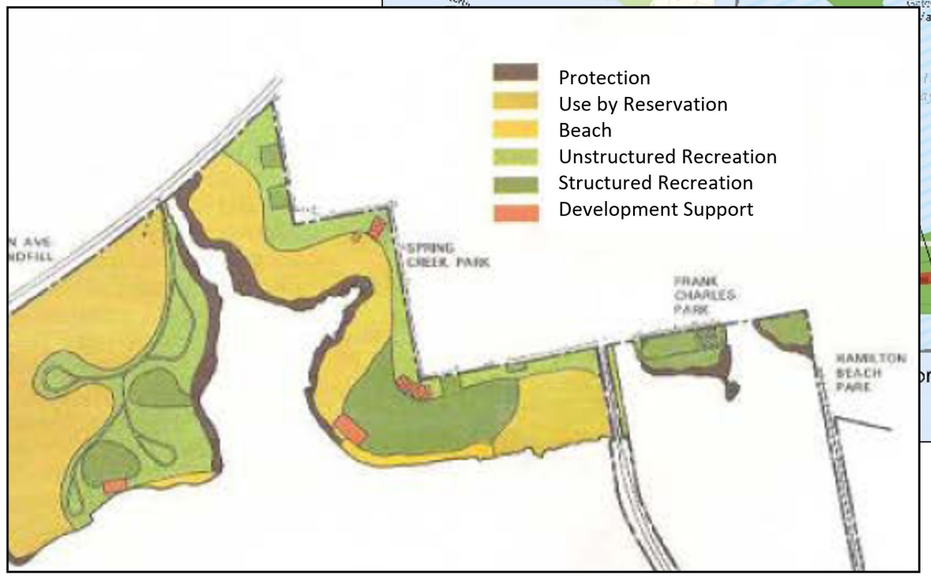
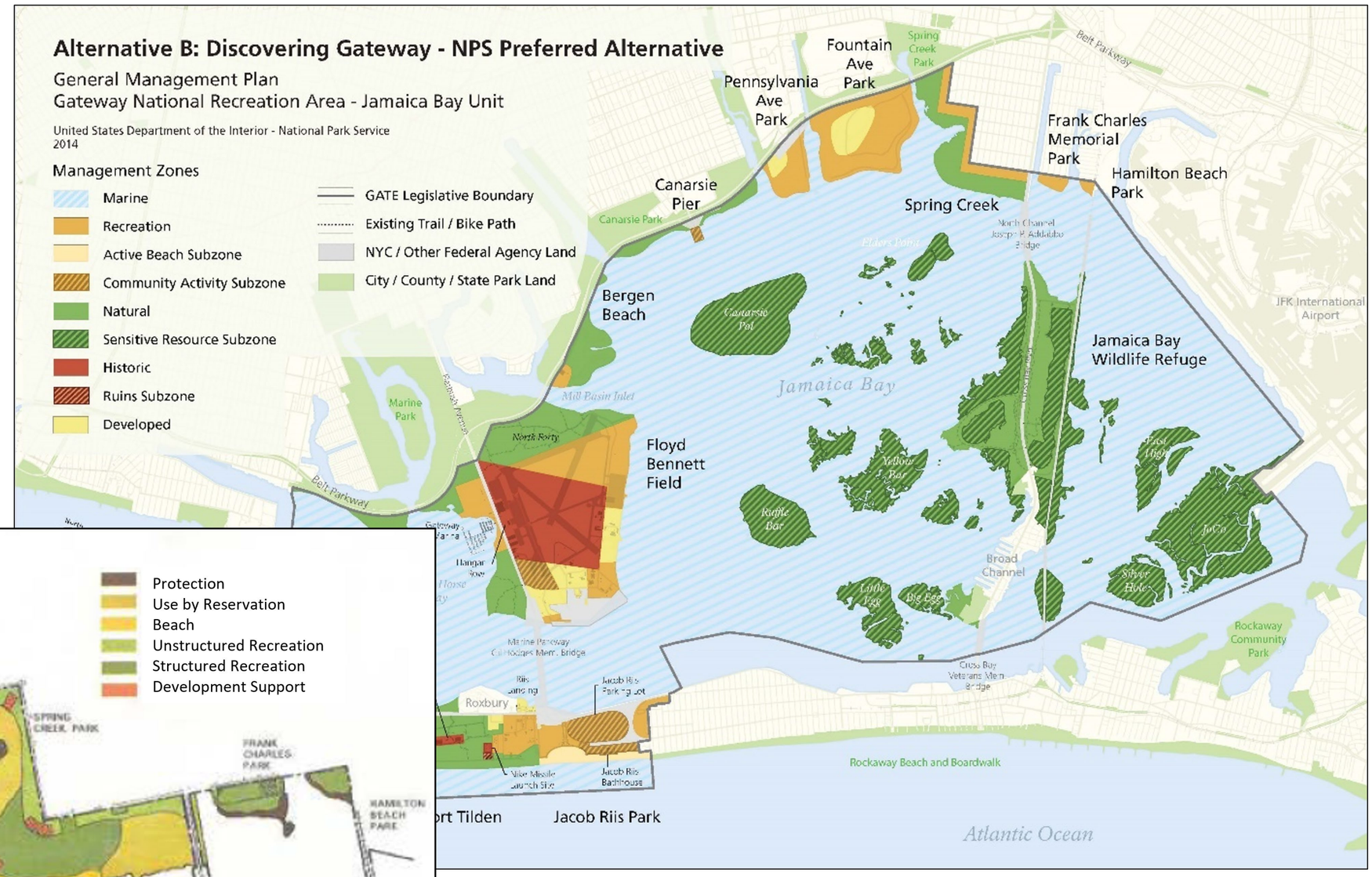


Figure 2-8. Aerial Imagery of Spring Creek Park 1975, 1980, 1985, 1994



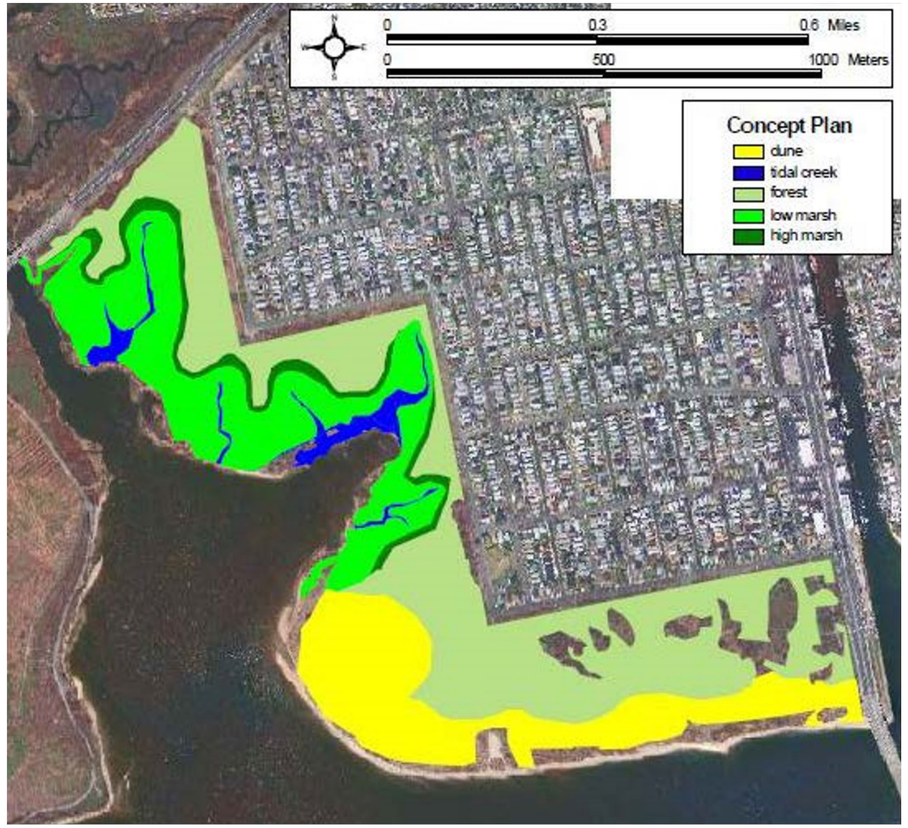
1979 General Management Plan
North Shore Management Zones (NPS, 1979)



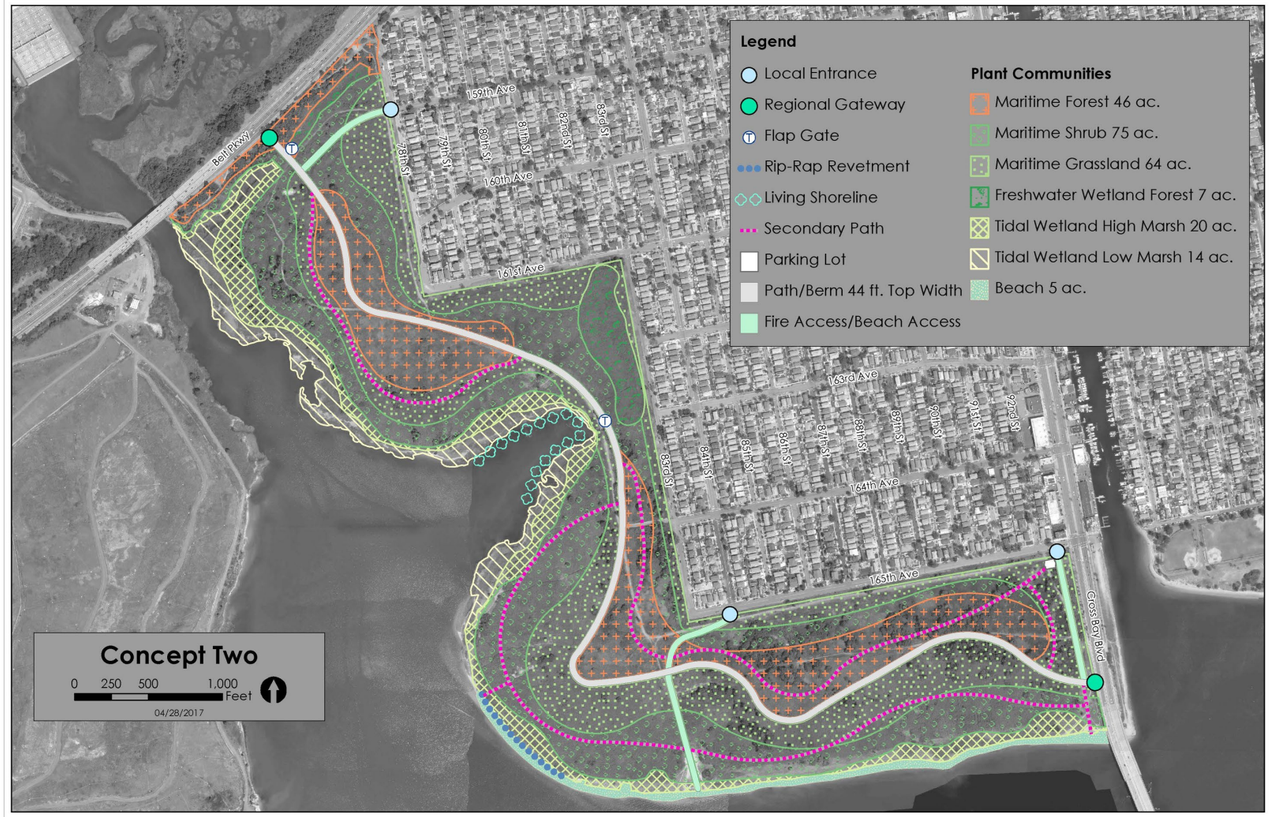
2014 General Management Plan/Environmental Impact Statement
Favored Alternative B: Discovering Gateway
Jamaica Bay Unit Management Zones (NPS, 2014b)

Figure 2-9. GMP Maps for the Jamaica Bay Unit Showing NPS Management Zones for Spring Creek Park

Figure 2-10. Aerial Imagery of Spring Creek Park 2004, 2008, 2017, 2020



Above: Feasibility Study recommended alternative concept plan for ecological restoration at Spring Creek Park. (USACE, 2013)



Above: Revised favored concept for ecological restoration incorporating coastal storm risk management features. (USACE, 2017b)

Right: Rendering of specific features of the revised favored concept. (Scape Studio, 2021)

Figure 2-11. Past Proposed Ecological Restoration Plans for Spring Creek Park

Figure 2-12. Historical Gamma Survey Coverage

Figure 2-13. Past Sampling Locations at Spring Creek Park

Figure 2-14. Map showing 2014 GMP/EIS NPS Management Zones and Site Decision Units at Spring Creek Park

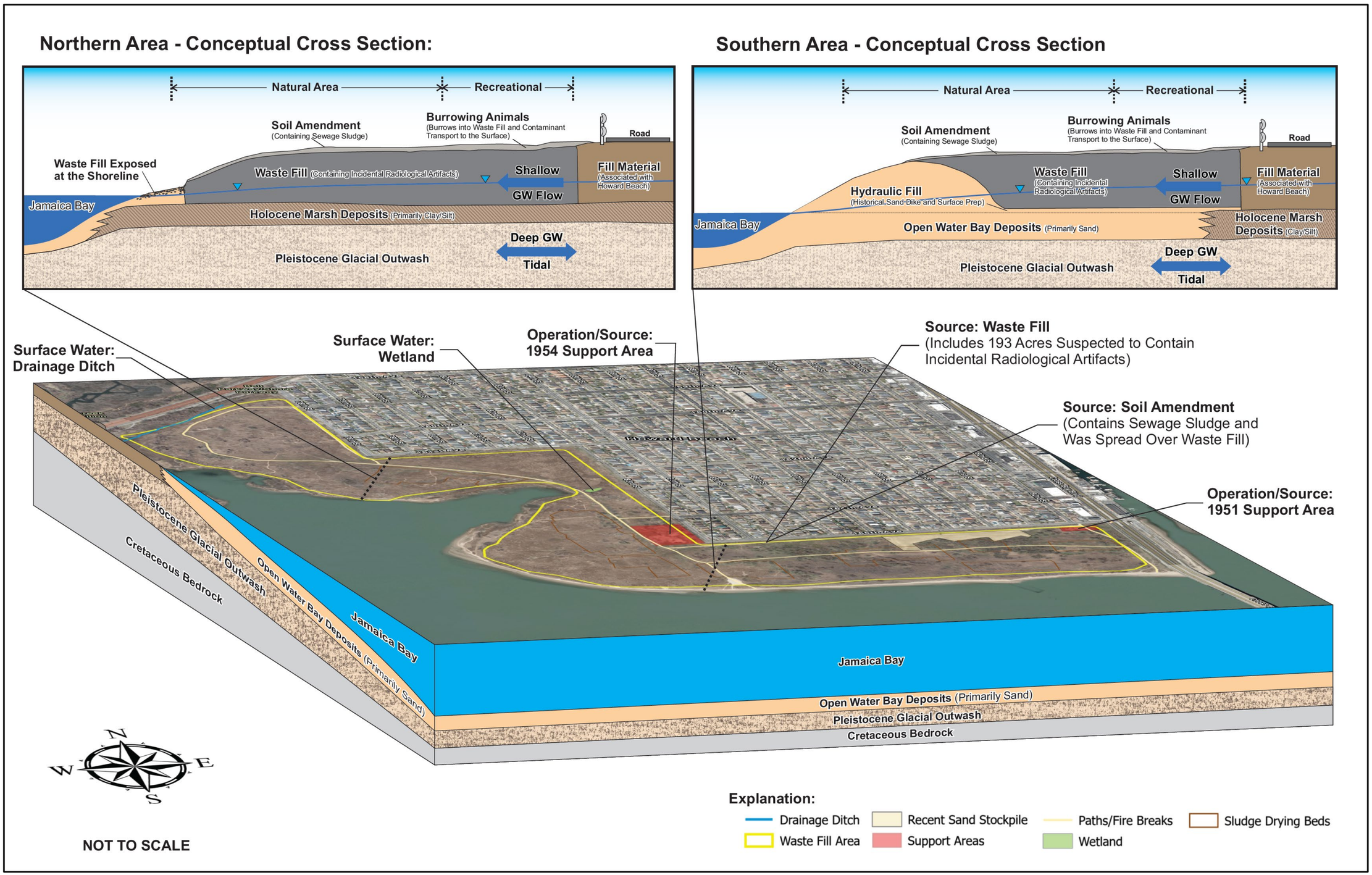
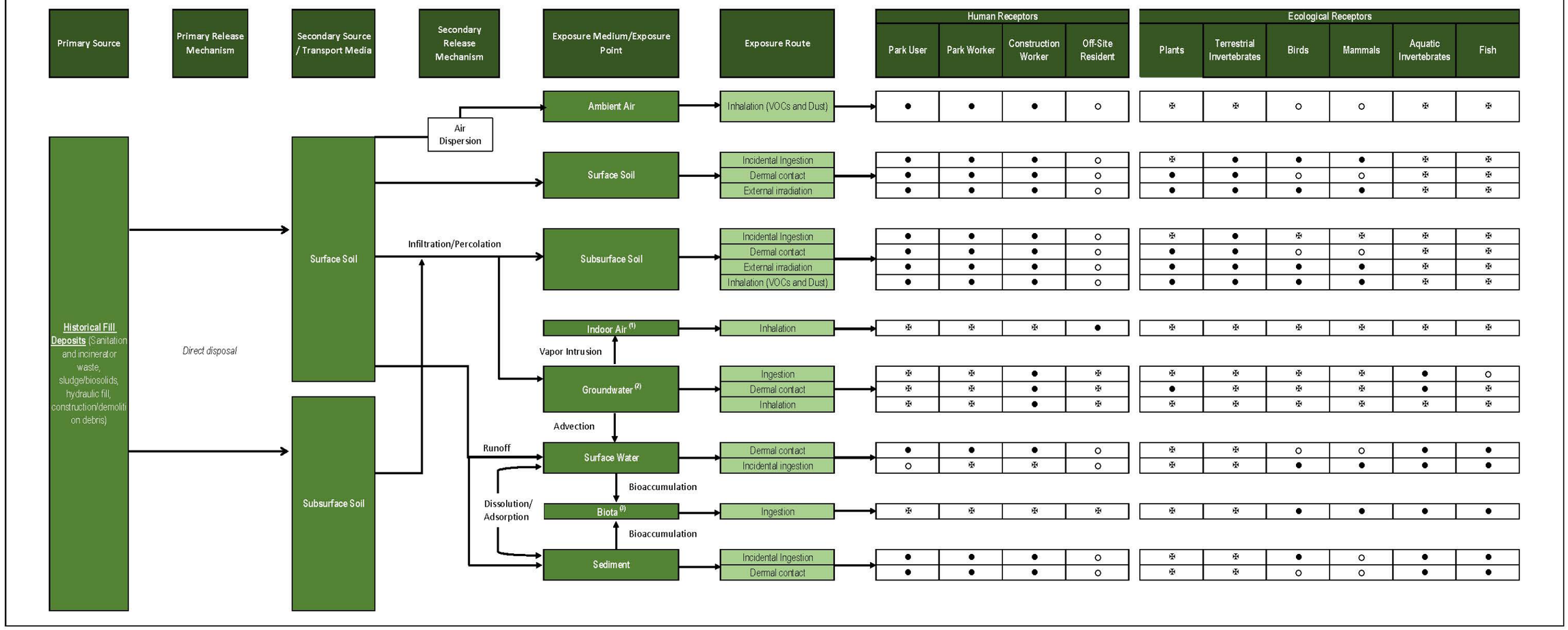


Figure 2-15. Graphical Conceptual Site Model for the Spring Creek Park Site



LEGEND:

- → Expected/potential transport pathway
- ● Complete exposure pathway
- ○ Potential exposure pathway
- ✠ Not a relevant exposure pathway

NOTES:

VOCs = volatile organic compounds.

1. Vapor intrusion will only be considered a viable pathway if VOCs are detected in shallow groundwater (<10 feet below grade) near the Site perimeter by the adjacent neighborhood.
2. For ecological receptors, shallow groundwater is considered a surrogate for sediment pore water exposure.
3. While it is possible that fishing could occur in Jamaica Bay, there is a fish advisory for the bay. Wetlands located within the footprint of the landfill are generally small and shallow and are not considered significant for recreational fishing. Thus, the fish ingestion pathway is considered incomplete for human receptors.

Additionally, a park user is not expected to be exposed through ingestion of wild or farmed foods. As the Site is located in an urban setting within a National Recreational Area, the land is not used and will not be used for hunting or farming (including community gardens). Collection of wild foods is not anticipated to occur to any appreciable degree.

Figure 2-16. Preliminary Pathway Receptor Diagram

Figure 4-1. Site Map Planned Sampling Locations

Tables

Table 4-1. Summary of Recovered Artifacts

Article	Dose	Details
5 Deck Markers	5 to 6 mR/hr on contact	Depth of burial 3" to 12" below ground surface 
1 Clip	0.7 mR/hr on contact	Depth of burial 6" 

Table 4-2. Summary of Data to be Collected

Data Description	How the Data Will Be Used
Soil samples analyzed for VOCs, SVOCs, PAHs, metals (including mercury and hexavalent chromium), herbicides, pesticides, PCBs,	Evaluate risks to human health and the environment, characterize the nature and extent of contamination in soil, and evaluate the fate and transport of contaminants in soil.
Groundwater samples analyzed for VOCs, SVOCs, PAHs, metals (including mercury and hexavalent chromium), herbicides, pesticides, PCBs, dioxins/furans, and radionuclide.	Evaluate risks to human health, characterize the nature and extent of contamination in groundwater, and evaluate the fate and transport of contaminants in groundwater.
Sediment analyzed for VOCs, SVOCs, PAHs, metals (including mercury and hexavalent), herbicides, pesticides, PCBs, dioxins/furans, and radionuclide.	Evaluate risks to human health and the environment, characterize the nature and extent of contamination in Site drainage channels, and evaluate the fate and transport of Site contaminants within drainage channels.
Soil gas samples analyzed for VOCs.	Assess the potential for VOC migration into the adjacent neighborhood and evaluate potential risks to human health as a result of vapor intrusion into indoor air.
Field screening of soil gas using a portable combustible gas indicator to determine the presence of CH_4, CO_2, O_2, CO, and H_2S.	Evaluate the Site for the presence of landfill gases and determine whether mitigation is required.
Results of gamma walkover surveys along established firebreaks (including a 10-ft buffer) and within 100-ft by 100-ft areas around the 105 soil sampling locations.	Evaluate the Site for the presence elevated radioactivity at the ground surface to support the selection of radiological anomalies and/or anomaly areas to be included in the focused investigations in waste fill.
Results of focused investigations of radiological anomalies in waste fill, including soil analytical results, count rates, and dose rates (waist level and contact).	Conduct a dose assessment and characterize the nature and extent of radiological artifacts and/or radiological soil contamination within waste fill.
Visual and field screening instrumentation readings during processing of soil borings.	Determine the screened intervals for groundwater monitoring wells, develop soil boring logs, assess the vertical extent of waste fill in OU2, and determine the interval of subsurface soil samples.
Other water quality parameters (e.g., temperature, conductivity, pH, ORP, salinity, dissolved oxygen, and turbidity) will be measured in the field during groundwater and surface water sampling.	Determine the point at which groundwater parameters have stabilized so the well can be sampled and provide groundwater geochemical information to aide in interpreting analytical data.
Land Survey information.	Geo-reference the activities listed above.

Table 4-3. Summary of Planned Site Sampling Activities

Location ID	Rationale
Surface Soil	
SCP-DPT-001 to SCP-DPT-105	Evaluate risks to human health and the environment, characterize the nature and extent of contamination, and evaluate fate and transport of contaminants for soil in waste filled areas of the Site.
SCP-DPT-106 to SCP-DPT-107	Characterize the nature of surface soil contamination in the waste disposal operational support areas shown on the 1951 and 1954 aerial photographs.
Subsurface Soil	
SCP-DPT-001 to SCP-DPT-105	Evaluate risks to human health and the environment, characterize the nature and extent of contamination, and evaluate fate and transport of contaminants for soil in waste filled areas of the Site.
SCP-DPT-106 to SCP-DPT-107	Characterize the nature of subsurface soil contamination in the waste disposal operational support areas shown on the 1951 and 1954 aerial photographs.
Groundwater	
SCP-MW-22 and SCP-MW-23	Characterize the nature of shallow groundwater contamination underlying the waste disposal operational support areas shown on the 1951 and 1954 aerial photographs.
Surface Water and Sediment	
SCP-SED-001 to SCP-SED-005	Evaluate risks to human health and the environment, characterize the nature and extent of contamination, and evaluate the fate and transport of Site contaminants in sediment present in Site surface water features.
Soil Gas	
SCP-SG-001 to SCP-SG-010	Assess the potential for VOC migration into the adjacent neighborhood and evaluate potential risks to human health as a result of vapor intrusion into indoor air.

Table 4-4. SAP Reference Limits for Soil

Chemical	Method	CAS #	Unit	Screening Benchmarks				Laboratory Limits	
				EPA Res. RSL (2021, HQ=0.1)	EPA Ind. RSL (2021, HQ=0.1)	Plant and Invert. ESV	Bird and Mammal ESV	LOQ	LOD
Pesticides									
4,4'-DDD	8081B	72-54-8	mg/kg	0.19	2.5	NE	0.0063	0.0017	0.0012
4,4'-DDE	8081B	72-55-9	mg/kg	2	9.3	NE	0.021	0.0017	0.0012
4,4'-DDT	8081B	50-29-3	mg/kg	1.9	8.5	4.1	0.021	0.0017	0.0016
Aldrin	8081B	309-00-2	mg/kg	0.039	0.18	0.00332	0.037	0.00083	0.0006
alpha Chlordane	8081B	5103-71-9	mg/kg	3.6	50	2.2	0.27	0.00083	0.0006
alpha-BHC	8081B	319-84-6	mg/kg	0.086	0.36	NE	0.1	0.00083	0.0006
beta-BHC	8081B	319-85-7	mg/kg	0.3	1.3	0.00398	NE	0.001	0.0009
Chlordane	8081B	12789-03-6	mg/kg	1.7	7.7	0.224	0.27	0.017	0.008
delta-BHC	8081B	319-86-8	mg/kg	NE	NE	NE	0.1	0.001	0.0009
Dieldrin	8081B	60-57-1	mg/kg	0.034	0.14	10	NE	0.0017	0.0012
Endosulfan I	8081B	959-98-8	mg/kg	NE	NE	NE	0.64	0.00083	0.0006
Endosulfan II	8081B	33213-65-9	mg/kg	NE	NE	NE	0.64	0.0023	0.0022
Endosulfan sulfate	8081B	1031-07-8	mg/kg	38	490	NE	0.64	0.0017	0.0012
Endrin	8081B	72-20-8	mg/kg	1.9	25	0.0034	0.0014	0.0017	0.0012
Endrin aldehyde	8081B	7421-93-4	mg/kg	NE	NE	NE	NE	0.0017	0.0012
Endrin ketone	8081B	53494-70-5	mg/kg	NE	NE	NE	NE	0.002	0.0018
gamma-Chlordane	8081B	5103-74-2	mg/kg	NE	NE	2.2	2.2	0.00083	0.0006
gamma-BHC (Lindane)	8081B	58-89-9	mg/kg	0.57	2.5	0.005	0.0094	0.00083	0.0006
Heptachlor	8081B	76-44-8	mg/kg	0.13	0.63	0.4	0.059	0.00083	0.00062
Heptachlor epoxide	8081B	1024-57-3	mg/kg	0.07	0.33	NE	NE	0.00083	0.0006
Methoxychlor	8081B	72-43-5	mg/kg	32	410	NE	5	0.0067	0.0065
Toxaphene	8081B	8001-35-2	mg/kg	0.49	2.1	NE	4.1	0.033	0.028
PCBs									
Aroclor 1016	8082A	12674-11-2	mg/kg	0.41	5.1	NE	1	0.017	0.01
Aroclor 1221	8082A	11104-28-2	mg/kg	0.2	0.83	NE	NE	0.017	0.01
Aroclor 1232	8082A	11141-16-5	mg/kg	0.17	0.72	NE	NE	0.017	0.01
Aroclor 1242	8082A	53469-21-9	mg/kg	0.23	0.95	NE	0.041	0.017	0.01
Aroclor 1248	8082A	12672-29-6	mg/kg	0.23	0.94	NE	0.0072	0.017	0.01
Aroclor 1254	8082A	11097-69-1	mg/kg	0.12	0.97	160	0.041	0.017	0.01
Aroclor 1260	8082A	11096-82-5	mg/kg	0.24	0.99	NE	0.14	0.017	0.01
Aroclor 1262	8082A	37324-23-5	mg/kg	NE	NE	NE	NE	0.017	0.01
Aroclor 1268	8082A	11100-14-4	mg/kg	NE	NE	NE	NE	0.017	0.01
Herbicides									
2,4,5-T	8151A	93-76-5	mg/kg	63	820	NE	NE	0.0017	0.00164
2,4,5-TP (Silvex)	8151A	93-72-1	mg/kg	51	660	NE	NE	0.0017	0.0015
2,4-D	8151A	94-75-7	mg/kg	70	960	NE	NE	0.036	0.024
2,4-DB	8151A	94-82-6	mg/kg	190	2500	NE	NE	0.021	.0.02
Dalapon	8151A	75-99-0	mg/kg	190	2500	NE	NE	0.09	0.088
Dicamba	8151A	1918-00-9	mg/kg	190	2500	NE	NE	0.012	0.008
Dichlorprop	8151A	120-36-5	mg/kg	NE	NE	NE	NE	0.02	0.018
Dinoseb	8151A	88-85-7	mg/kg	6.3	82	NE	NE	0.024	0.018
MCPA (2-Methyl-4-chlorophenoxy acetic acid)	8151A	94-74-6	mg/kg	3.2	41	NE	NE	2.5	1.52
MCPP (2-(2-Methyl-4-chlorophenoxy) propanoic acid)	8151A	93-65-2	mg/kg	6.3	82	NE	NE	7.7	7.6
VOCs									
1,1,1-Trichloroethane	8260C	71-55-6	mg/kg	810	3600	260	NE	0.0005	0.0002
1,1,2,2-Tetrachloroethane	8260C	79-34-5	mg/kg	0.6	2.7	NE	NE	0.005	0.001
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	8260C	76-13-1	mg/kg	670	2800	NE	NE	0.01	0.002
1,1,2-Trichloroethane	8260C	79-00-5	mg/kg	0.15	0.63	NE	NE	0.005	0.002
1,1-Dichloroethane	8260C	75-34-3	mg/kg	3.6	16	NE	210	0.005	0.002
1,1-Dichloroethene	8260C	75-35-4	mg/kg	23	100	NE	11	0.005	0.002
1,2,4-Trichlorobenzene	8260C	120-82-1	mg/kg	5.8	26	1.2	0.27	0.01	0.008
1,2-Dibromo-3-chloropropane	8260C	96-12-8	mg/kg	0.0053	0.064	NE	NE	0.005	0.001
1,2-Dibromoethane	8260C	106-93-4	mg/kg	0.036	0.16	NE	NE	0.005	0.001
1,2-Dichlorobenzene	8260C	95-50-1	mg/kg	180	930	20	0.92	0.005	0.002
1,2-Dichloroethane	8260C	107-06-2	mg/kg	0.46	2	NE	0.85	0.005	0.002
1,2-Dichloropropane	8260C	78-87-5	mg/kg	1.6	6.6	NE	NE	0.005	0.002
1,3-Dichlorobenzene	8260C	541-73-1	mg/kg	NE	NE	NE	0.73	0.005	0.002
1,4-Dichlorobenzene	8260C	106-46-7	mg/kg	2.6	11	NE	0.88	0.005	0.001
2-Butanone	8260C	78-93-3	mg/kg	2700	19000	NE	NE	0.01	0.004
2-Hexanone	8260C	591-78-6	mg/kg	20	130	NE	0.36	0.01	0.004
4-Methyl-2-pentanone	8260C	108-10-1	mg/kg	3300	14000	NE	9.8	0.01	0.004
Acetone	8260C	67-64-1	mg/kg	6100	67000	NE	1.2	0.02	0.016
Benzene	8260C	71-43-2	mg/kg	1.2	5.1	NE	24	0.005	0.002
Bromodichloromethane	8260C	75-27-4	mg/kg	0.29	1.3	NE	NE	0.005	0.001
Bromoform	8260C	75-25-2	mg/kg	19	86	NE	NE	0.01	0.008
Bromomethane	8260C	74-83-9	mg/kg	0.68	3	NE	NE	0.005	0.002
Carbon disulfide	8260C	75-15-0	mg/kg	77	350	NE	0.82	0.005	0.002
Carbon tetrachloride	8260C	56-23-5	mg/kg	0.65	2.9	NE	58.6	0.005	0.002
Chlorobenzene	8260C	108-90-7	mg/kg	28	130	2.4	43	0.005	0.002
Chloroethane	8260C	75-00-3	mg/kg	1400	5700	NE	NE	0.005	0.004
Chloroform	8260C	67-66-3	mg/kg	0.32	1.4	NE	8	0.005	0.002
Chloromethane	8260C	74-87-3	mg/kg	11	46	NE	NE	0.005	0.002
cis-1,2-Dichloroethene	8260C	156-59-2	mg/kg	16	230	NE	89.6	0.005	0.002
cis-1,3-Dichloropropene	8260C	10061-01-5	mg/kg	NE	NE	NE	NE	0.005	0.001
cyclohexane	8260C	110-82-7	mg/kg	650	2700	NE	NE	0.005	0.002
Cyclohexanone	8260C	108-94-1	mg/kg	2800	13000	NE	NE	12.5	5
Dibromochloromethane	8260C	124-48-1	mg/kg	8.3	39	NE	NE	0.005	0.001
Dichlorodifluoromethane	8260C	75-71-8	mg/kg	8.7	37	NE	NE	0.005	0.002
Ethylbenzene	8260C	100-41-4	mg/kg	5.8	25	NE	NE	0.005	0.001
Isopropylbenzene	8260C	98-82-8	mg/kg	190	990	NE	NE	0.005	0.001
Methylacetate	8260C	79-20-9	mg/kg	7800	120000	NE	NE	0.005	0.004
Methyl tert-butyl ether	8260C	1634-04-4	mg/kg	47	210	NE	NE	0.005	0.002
methylcyclohexane	8260C	108-87-2	mg/kg	NE	NE	NE	NE	0.005	0.002
Methylene chloride	8260C	75-09-2	mg/kg	35	320	1600	2.6	0.005	0.004

Table 4-4. SAP Reference Limits for Soil

Chemical	Method	CAS #	Unit	Screening Benchmarks				Laboratory Limits	
				EPA Res. RSL (2021, HQ=0.1)	EPA Ind. RSL (2021, HQ=0.1)	Plant and Invert. ESV	Bird and Mammal ESV	LOQ	LOD
Styrene	8260C	100-42-5	mg/kg	600	3500	1.2	NE	0.005	0.001
Tetrachloroethene	8260C	127-18-4	mg/kg	8.1	39	10	0.18	0.005	0.002
Toluene	8260C	108-88-3	mg/kg	490	4700	200	23	0.005	0.002
trans-1,2-Dichloroethene	8260C	156-60-5	mg/kg	7	30	NE	89.6	0.005	0.002
trans-1,3-Dichloropropene	8260C	10061-02-6	mg/kg	NE	NE	NE	NE	0.005	0.001
Trichloroethene	8260C	79-01-6	mg/kg	0.41	1.9	NE	1.387	0.005	0.002
Trichlorofluoromethane	8260C	75-69-4	mg/kg	2300	35000	NE	52	0.005	0.002
Vinyl chloride	8260C	75-01-4	mg/kg	0.059	1.7	NE	0.12	0.005	0.002
Xylenes (total)	8260C	1330-20-7	mg/kg	58	250	10	1.4	0.01	0.002
SVOCs									
1,2,4-Trichlorobenzene	8270D	120-82-1	mg/kg	5.8	26	1.2	0.27	0.0367	0.0333
1,2-Dichlorobenzene	8270D	95-50-1	mg/kg	180	930	20	0.92	0.0367	0.0333
1,3-Dichlorobenzene	8270D	541-73-1	mg/kg	NE	NE	NE	0.73	0.0367	0.0333
1,4-Dichlorobenzene	8270D	106-46-7	mg/kg	2.6	11	NE	0.88	0.0367	0.0333
1,4-Dioxane	8270D	123-91-1	mg/kg	5.3	24	NE	NE	0.167	0.0667
2,4,5-Trichlorophenol	8270D	95-95-4	mg/kg	630	8200	4	NE	0.0367	0.0333
2,4,6-Trichlorophenol	8270D	88-06-2	mg/kg	6.3	82	10	NE	0.0367	0.0333
2,4-Dichlorophenol	8270D	120-83-2	mg/kg	19	250	NE	NE	0.0433	0.04
2,4-Dimethylphenol	8270D	105-67-9	mg/kg	130	1600	0.01	NE	0.0367	0.0333
2,4-Dinitrophenol	8270D	51-28-5	mg/kg	13	160	20	NE	1	0.333
2,4-Dinitrotoluene	8270D	121-14-2	mg/kg	1.7	7.4	NE	2.5	0.167	0.0667
2,6-Dinitrotoluene	8270D	606-20-2	mg/kg	0.36	1.5	NE	1.8	0.0367	0.0333
2-Chloronaphthalene	8270D	91-58-7	mg/kg	480	6000	NE	NE	0.0333	0.0267
2-Chlorophenol	8270D	95-57-8	mg/kg	39	580	NE	0.39	0.0367	0.0333
2-Methylnaphthalene	8270D	91-57-6	mg/kg	24	300	NE	16	0.0167	0.00999
2-Methylphenol	8270D	95-48-7	mg/kg	320	4100	0.67	590	0.05	0.04
2-Nitroaniline	8270D	88-74-4	mg/kg	63	800	NE	5.4	0.05	0.0333
2-Nitrophenol	8270D	88-75-5	mg/kg	NE	NE	7	NE	0.05	0.04
3,3-Dichlorobenzidine	8270D	91-94-1	mg/kg	1.2	5.1	NE	NE	0.167	0.0667
3-Nitroaniline	8270D	99-09-2	mg/kg	NE	NE	NE	NE	0.167	0.0667
4,6-Dinitro-2-methylphenol	8270D	534-52-1	mg/kg	0.51	6.6	NE	NE	0.5	0.333
4-Bromophenyl-phenylether	8270D	101-55-3	mg/kg	NE	NE	NE	NE	0.0367	0.0333
4-Chloro-3-methylphenol	8270D	59-50-7	mg/kg	630	8200	NE	NE	0.05	0.04
4-Chloroaniline	8270D	106-47-8	mg/kg	2.7	11	1	NE	0.167	0.0667
4-Chlorophenyl-phenylether	8270D	7005-72-3	mg/kg	NE	NE	NE	NE	0.0367	0.0333
4-Nitroaniline	8270D	100-01-6	mg/kg	25	110	NE	NE	0.167	0.0667
4-Nitrophenol	8270D	100-02-7	mg/kg	NE	NE	7	NE	0.5	0.333
Aniline	8270D	62-53-3	mg/kg	44	400	NE	NE	0.167	0.0667
Benzyl Alcohol	8270D	100-51-6	mg/kg	630	8200	NE	NE	0.5	0.333
Bis(2-chloro-1-methylethyl) ether	8270D	108-60-1	mg/kg	310	4700	NE	NE	0.0433	0.04
Bis(2-chloroethoxy)methane	8270D	111-91-1	mg/kg	19	250	NE	NE	0.0367	0.0333
Bis(2-chloroethyl)ether	8270D	111-44-4	mg/kg	0.23	1	NE	NE	0.0367	0.0333
Bis(2-ethylhexyl)phthalate	8270D	117-81-7	mg/kg	39	160	NE	0.02	0.167	0.133
CARBAZOLE	8270D	86-74-8	mg/kg	NE	NE	NE	80	0.0367	0.0333
Dibenzofuran	8270D	132-64-9	mg/kg	7.8	120	6.1	NE	0.0367	0.0333
Diethyl phthalate	8270D	84-66-2	mg/kg	5100	66000	100	3600	0.167	0.133
Dimethyl phthalate	8270D	131-11-3	mg/kg	NE	NE	10	38	0.167	0.133
Di-n-butyl phthalate	8270D	84-74-2	mg/kg	630	8200	160	0.011	0.167	0.133
Di-n-octyl phthalate	8270D	117-84-0	mg/kg	63	820	NE	0.91	0.167	0.133
Hexachlorobenzene	8270D	118-74-1	mg/kg	0.21	0.96	10	0.079	0.0167	0.0133
Hexachlorobutadiene	8270D	87-68-3	mg/kg	1.2	5.3	NE	NE	0.05	0.04
hexachlorocyclopentadiene	8270D	77-47-4	mg/kg	0.18	0.75	10	NE	0.5	0.333
Hexachloroethane	8270D	67-72-1	mg/kg	1.8	8	NE	NE	0.167	0.0667
Isophorone	8270D	78-59-1	mg/kg	570	2400	NE	NE	0.0667	0.0333
Nitrobenzene	8270D	98-95-3	mg/kg	5.1	22	2.2	4.9	0.0367	0.0333
n-Nitroso-di-n-propylamine	8270D	621-64-7	mg/kg	0.078	0.33	NE	NE	0.0667	0.05
n-Nitrosodiphenylamine	8270D	86-30-6	mg/kg	110	470	20	NE	0.0367	0.0333
Pentachlorophenol	8270D	87-86-5	mg/kg	1	4	3	0.36	0.167	0.133
Phenol	8270D	108-95-2	mg/kg	1900	25000	0.79	38	0.0367	0.0333
Pyridine	8270D	110-86-1	mg/kg	7.8	120	NE	NE	0.167	0.133
PAHs									
Acenaphthene	8270D-SIM	83-32-9	mg/kg	360	4500	0.25	120	0.00167	0.00133
Acenaphthylene	8270D-SIM	208-96-8	mg/kg	NE	NE	NE	120	0.00167	0.00133
Anthracene	8270D-SIM	120-12-7	mg/kg	1800	23000	6.8	210	0.00167	0.00133
Benzo(a)anthracene	8270D-SIM	56-55-3	mg/kg	1.1	21	18	0.8	0.00167	0.00133
Benzo(a)pyrene	8270D-SIM	50-32-8	mg/kg	0.11	2.1	NE	53	0.00167	0.00133
Benzo(b)fluoranthene	8270D-SIM	205-99-2	mg/kg	1.1	21	18	38	0.00167	0.00133
Benzo(g,h,i)perylene	8270D-SIM	191-24-2	mg/kg	NE	NE	NE	1.98	0.00167	0.00133
Benzo(k)fluoranthene	8270D-SIM	207-08-9	mg/kg	11	210	NE	62	0.00167	0.00133
Chrysene	8270D-SIM	218-01-9	mg/kg	110	2100	2.4	NE	0.00167	0.00133
Dibenz(a,h)anthracene	8270D-SIM	53-70-3	mg/kg	0.11	2.1	NE	12	0.00167	0.00133
Fluoranthene	8270D-SIM	206-44-0	mg/kg	240	3000	10	22	0.00167	0.00133
Fluorene	8270D-SIM	86-73-7	mg/kg	240	3000	3.7	250	0.00167	0.00133
Indeno(1,2,3-cd)pyrene	8270D-SIM	193-39-5	mg/kg	1.1	21	NE	62	0.00167	0.00133
Naphthalene	8270D-SIM	91-20-3	mg/kg	2	8.6	1	3.4	0.00333	0.00267
Phenanthrene	8270D-SIM	85-01-8	mg/kg	NE	NE	5.5	10	0.00233	0.002
Pyrene	8270D-SIM	129-00-0	mg/kg	180	2300	10	22	0.00167	0.00133
Metals									
Aluminum	6020B	7429-90-5	mg/kg	7700	110000	50	NE	10	8
Antimony	6020B	7440-36-0	mg/kg	3.1	47	0.05	0.248	0.2	0.1
Arsenic	6020B	7440-38-2	mg/kg	0.68	3	6.8	0.25	0.2	0.16
Barium	6020B	7440-39-3	mg/kg	1500	22000	110	17.2	0.2	0.16
Beryllium	6020B	7440-41-7	mg/kg	16	230	2.5	2.42	0.5	0.025
Cadmium	6020B	7440-43-9	mg/kg	7.1	98	4	0.27	0.05	0.04
Calcium (Ca)	6020B	7440-70-2	mg/kg	NE	NE	NE	NE	25	16
Chromium	6020B	7440-47-3	mg/kg	NE	NE	0.35	28	0.4	0.16

Table 4-4. SAP Reference Limits for Soil

Chemical	Method	CAS #	Unit	Screening Benchmarks				Laboratory Limits	
				EPA Res. RSL (2021, HQ=0.1)	EPA Ind. RSL (2021, HQ=0.1)	Plant and Invert. ESV	Bird and Mammal ESV	LOQ	LOD
Cobalt	6020B	7440-48-4	mg/kg	2.3	35	13	96	0.1	0.05
Copper	6020B	7440-50-8	mg/kg	310	4700	50	15	0.2	0.16
Iron (Fe)	6020B	7439-89-6	mg/kg	5500	82000	NE	NE	10	10
Lead	6020B	7439-92-1	mg/kg	400	800	50	0.94	0.1	0.05
Magnesium (Mg)	6020B	7439-95-4	mg/kg	NE	NE	NE	NE	5	5
Manganese (Mn)	6020B	7439-96-5	mg/kg	NE	NE	220	322	0.2	0.16
Nickel	6020B	7440-02-0	mg/kg	150	2200	30	9.7	0.2	0.16
Potassium (K)	6020B	7440-09-7	mg/kg	NE	NE	NE	NE	20	16
Selenium	6020B	7782-49-2	mg/kg	39	580	NE	NE	0.2	0.1
Silver	6020B	7440-22-4	mg/kg	NE	NE	2	2.6	0.05	0.04
Sodium (Na)	6020B	7440-23-5	mg/kg	NE	NE	NE	NE	25	20
Thallium	6020B	7440-28-0	mg/kg	0.078	1.2	0.1	0.027	0.05	0.04
Vanadium	6020B	7440-62-2	mg/kg	39	580	0.025	0.714	0.4	0.1
Zinc	6020B	7440-66-6	mg/kg	2300	35000	6.62	12	15	0.75
Mercury	7471B	7439-97-6	mg/kg	1.1	4.6	0.05	0.013	0.083	0.066
Hexavalent Chromium	7199	18540-29-9	mg/kg	0.3	6.3	NE	12.01	0.4	0.3
Dioxins/Furans									
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	8290A	67562-39-4	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	8290A	35822-46-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	8290A	55673-89-7	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	70648-26-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	39227-28-6	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	57117-44-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	57653-85-7	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	8290A	72918-21-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	19408-74-3	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	8290A	57117-41-6	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	8290A	40321-76-4	mg/kg	NE	NE	NE	NE	0.000005	0.000004
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	60851-34-5	mg/kg	NE	NE	NE	NE	0.000005	0.000004
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	8290A	57117-31-4	mg/kg	NE	NE	NE	NE	0.000005	0.000004
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	8290A	51207-31-9	mg/kg	NE	NE	NE	NE	0.000001	0.0000008
Octachlorodibenzofuran (OCDF)	8290A	39001-02-0	mg/kg	NE	NE	NE	NE	0.00001	0.000008
Octachlorodibenzo-p-dioxin (OCDD)	8290A	3268-87-9	mg/kg	NE	NE	NE	NE	0.00001	0.000008
2,3,7,8-TCDD	8290A	1746-01-6	mg/kg	0.0000048	0.000022	NE	NE	0.000001	0.0000008
Radionuclides[1]									
Uranium-238 (via Th-234)	901.1	7440-61-1	pCi/g	0.00176[1]	14[2]	400	1100	1	NE
Radium-226 (via Bi-214)	901.1	14733-03-0	pCi/g	0.00182[1]	0.6[2][3]	54	1.5	1	NE
Thorium-232 (via Ac-228)	901.1	14331-83-0	pCi/g	0.00174[1]	1.1[2]	24	6.2	1	NE
Thorium-228 (Isotopic Thorium)	HASL 300	14274-82-9	pCi/g	0.00706[1]	4.7[2]	140	43	1	NE
Thorium-230 (Isotopic Thorium)	HASL 300	14269-63-7	pCi/g	0.00182[1]	1.8[2]	200	52	1	NE
Thorium-232 (Isotopic Thorium)	HASL 300	7440-29-1	pCi/g	0.00174[1]	1.1[2]	24	6.2	1	NE
Uranium-234 (Isotopic Uranium)	HASL 300	13966-29-5	pCi/g	0.00179[1]	1.3[2]	440	2200	1	NE
Uranium-235 (Isotopic Uranium)	HASL 300	15117-96-1	pCi/g	0.00623[1]	8[2]	440	1600	1	NE
Uranium-238 (Isotopic Uranium)	HASL 300	7440-61-1	pCi/g	0.00176[1]	14[2]	400	1100	1	NE
Gross Alpha	900	12587-46-1	pCi/g	NE	NE	NE	NE	10	NE
Gross Beta	900	12587-47-2	pCi/g	NE	NE	NE	NE	10	NE

Denotes a laboratory limit that exceeds one or more project screening benchmarks.

NE - Not Established

(1) Residential Preliminary Remediation Goals for Radionuclides calculated using the USEPA PRG calculator <https://epa-prgs.ornl.gov/radionuclides/>

(2) Human health soil screening benchmarks for radionuclides are based on soil screening levels for evaluating surface soil, as specified in US Nuclear Regulatory Commission Regulation (NUREG) 1757, Volume 1 (U.S. NRC, 2006).

(3) Screening level is based on Radium-226 in equilibrium with all its progeny.

Table 4-5. SAP Reference Limits for Groundwater

Chemical	Method	CAS #	Unit	Screening Benchmarks		Laboratory Limits	
				Tap Water RSLs (2021, HQ=0.1)	Federal MCLs	LOQ	LOD
Pesticides							
4,4'-DDD	8081B	72-54-8	ug/L	0.0063	NE	0.03	0.02
4,4'-DDE	8081B	72-55-9	ug/L	0.046	NE	0.03	0.02
4,4'-DDT	8081B	50-29-3	ug/L	0.23	NE	0.03	0.02
Aldrin	8081B	309-00-2	ug/L	0.00092	NE	0.02	0.01
alpha Chlordane	8081B	5103-71-9	ug/L	0.36	NE	0.02	0.01
alpha-BHC	8081B	319-84-6	ug/L	0.0072	NE	0.02	0.01
beta-BHC	8081B	319-85-7	ug/L	0.025	NE	0.02	0.01
Chlordane	8081B	12789-03-6	ug/L	0.02	2	0.5	0.32
delta-BHC	8081B	319-86-8	ug/L	NE	NE	0.02	0.01
Dieldrin	8081B	60-57-1	ug/L	0.0018	NE	0.03	0.02
Endosulfan I	8081B	959-98-8	ug/L	NE	NE	0.02	0.01
Endosulfan II	8081B	33213-65-9	ug/L	NE	NE	0.04	0.02
Endosulfan sulfate	8081B	1031-07-8	ug/L	11	NE	0.03	0.02
Endrin	8081B	72-20-8	ug/L	0.23	2	0.03	0.02
Endrin aldehyde	8081B	7421-93-4	ug/L	NE	NE	0.1	0.06
Endrin ketone	8081B	53494-70-5	ug/L	NE	NE	0.03	0.02
gamma-BHC (Lindane)	8081B	58-89-9	ug/L	0.042	0.2	0.02	0.01
gamma-Chlordane	8081B	5103-74-2	ug/L	NE	NE	0.02	0.02
Heptachlor	8081B	76-44-8	ug/L	0.0014	0.4	0.02	0.01
Heptachlor epoxide	8081B	1024-57-3	ug/L	0.0014	0.2	0.02	0.01
Methoxychlor	8081B	72-43-5	ug/L	3.7	40	0.11	0.1
Toxaphene	8081B	8001-35-2	ug/L	0.071	3	1	0.6
PCBs							
Aroclor 1016	8082A	12674-11-2	ug/L	0.14	NE	0.25	0.2
Aroclor 1221	8082A	11104-28-2	ug/L	0.0047	NE	0.25	0.2
Aroclor 1232	8082A	11141-16-5	ug/L	0.0047	NE	0.25	0.2
Aroclor 1242	8082A	53469-21-9	ug/L	0.0078	NE	0.25	0.2
Aroclor 1248	8082A	12672-29-6	ug/L	0.0078	NE	0.25	0.2
Aroclor 1254	8082A	11097-69-1	ug/L	0.0078	NE	0.25	0.2
Aroclor 1260	8082A	11096-82-5	ug/L	0.0078	NE	0.25	0.2
Aroclor 1262	8082A	37324-23-5	ug/L	NE	NE	0.25	0.2
Aroclor 1268	8082A	11100-14-4	ug/L	NE	NE	0.25	0.2
Herbicides							
2,4,5-T	8151A	93-76-5	ug/L	16	NE	0.15	0.13
2,4,5-TP (Silvex)	8151A	93-72-1	ug/L	11	50	0.05	0.03
2,4-D	8151A	94-75-7	ug/L	17	70	0.6	0.5
2,4-DB	8151A	94-82-6	ug/L	45	NE	1.5	1.3
Dalapon	8151A	75-99-0	ug/L	60	200	4	3.6
Dicamba	8151A	1918-00-9	ug/L	57	NE	0.3	0.16
Dichlorprop	8151A	120-36-5	ug/L	NE	NE	0.5	0.32
Dinoseb	8151A	88-85-7	ug/L	1.5	7	0.5	0.4
MCPA (2-Methyl-4-chlorophenoxy acetic acid)	8151A	94-74-6	ug/L	0.75	NE	200	100
MCPP (2-(2-Methyl-4-chlorophenoxy) propanoic acid)	8151A	93-65-2	ug/L	1.6	NE	200	100
VOCs							
1,1,1-Trichloroethane	8260C	71-55-6	ug/L	800	200	0.5	0.2
1,1,2,2-Tetrachloroethane	8260C	79-34-5	ug/L	0.076	NE	0.5	0.2
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	8260C	76-13-1	ug/L	1000	NE	0.5	0.2
1,1,2-Trichloroethane	8260C	79-00-5	ug/L	0.041	5	0.5	0.2
1,1-Dichloroethane	8260C	75-34-3	ug/L	2.8	NE	0.5	0.2
1,1-Dichloroethene	8260C	75-35-4	ug/L	28	7	0.5	0.2
1,2,4-Trichlorobenzene	8260C	120-82-1	ug/L	0.4	70	0.5	0.2
1,2-Dibromo-3-chloropropane	8260C	96-12-8	ug/L	0.00033	0.2	0.5	0.4
1,2-Dibromoethane	8260C	106-93-4	ug/L	0.0075	0.05	0.5	0.2
1,2-Dichlorobenzene	8260C	95-50-1	ug/L	30	600	0.5	0.2
1,2-Dichloroethane	8260C	107-06-2	ug/L	0.17	5	0.5	0.2
1,2-Dichloropropane	8260C	78-87-5	ug/L	0.82	5	0.5	0.2
1,3-Dichlorobenzene	8260C	541-73-1	ug/L	NE	NE	0.5	0.2
1,4-Dichlorobenzene	8260C	106-46-7	ug/L	0.48	75	0.5	0.2
2-Butanone	8260C	78-93-3	ug/L	560	NE	5	2
2-Hexanone	8260C	591-78-6	ug/L	3.8	NE	5	2
4-Methyl-2-pentanone	8260C	108-10-1	ug/L	630	NE	5	2
Acetone	8260C	67-64-1	ug/L	1400	NE	5	2
Benzene	8260C	71-43-2	ug/L	0.46	5	0.5	0.2
Bromodichloromethane	8260C	75-27-4	ug/L	0.13	NE	0.5	0.2
Bromoform	8260C	75-25-2	ug/L	3.3	NE	1	0.5
Bromomethane	8260C	74-83-9	ug/L	0.75	NE	0.5	0.2
Carbon disulfide	8260C	75-15-0	ug/L	81	NE	1	0.2
Carbon tetrachloride	8260C	56-23-5	ug/L	0.46	5	0.5	0.2
Chlorobenzene	8260C	108-90-7	ug/L	7.8	100	0.5	0.2
Chloroethane	8260C	75-00-3	ug/L	NE	NE	0.5	0.2
Chloroform	8260C	67-66-3	ug/L	0.22	NE	0.5	0.2
Chloromethane	8260C	74-87-3	ug/L	19	NE	0.5	0.2
cis-1,2-Dichloroethene	8260C	156-59-2	ug/L	3.6	70	0.5	0.2
cis-1,3-Dichloropropene	8260C	10061-01-5	ug/L	NE	NE	0.5	0.2
cyclohexane	8260C	110-82-7	ug/L	1300	NE	0.5	0.2
Cyclohexanone	8260C	108-94-1	ug/L	140	NE	25	7.2
Dibromochloromethane	8260C	124-48-1	ug/L	0.87	NE	0.5	0.2
Dichlorodifluoromethane	8260C	75-71-8	ug/L	20	NE	0.5	0.2
Ethylbenzene	8260C	100-41-4	ug/L	1.5	700	0.5	0.2
Isopropylbenzene	8260C	98-82-8	ug/L	45	NE	0.5	0.2
Methyl tert-butyl ether	8260C	1634-04-4	ug/L	14	NE	0.5	0.2
Methylacetate	8260C	79-20-9	ug/L	2000	NE	1	0.2
methylcyclohexane	8260C	108-87-2	ug/L	NE	NE	0.5	0.2
Methylene chloride	8260C	75-09-2	ug/L	11	5	0.5	0.2
Styrene	8260C	100-42-5	ug/L	120	100	0.5	0.2
Tetrachloroethene	8260C	127-18-4	ug/L	4.1	5	0.5	0.2

Table 4-5. SAP Reference Limits for Groundwater

Chemical	Method	CAS #	Unit	Screening Benchmarks		Laboratory Limits	
				Tap Water RSLs (2021, HQ=0.1)	Federal MCLs	LOQ	LOD
Toluene	8260C	108-88-3	ug/L	110	1000	0.5	0.2
trans-1,2-Dichloroethene	8260C	156-60-5	ug/L	6.8	100	0.5	0.2
trans-1,3-Dichloropropene	8260C	10061-02-6	ug/L	NE	NE	0.5	0.2
Trichloroethene	8260C	79-01-6	ug/L	0.28	5	0.5	0.2
Trichlorofluoromethane	8260C	75-69-4	ug/L	520	NE	0.5	0.2
Vinyl chloride	8260C	75-01-4	ug/L	0.019	2	0.5	0.2
Xylenes (total)	8260C	1330-20-7	ug/L	19	10000	1	0.4
SVOCs							
1,2,4-Trichlorobenzene	8270D	120-82-1	ug/L	0.4	70	2	1
1,2-Dichlorobenzene	8270D	95-50-1	ug/L	30	600	2	1
1,3-Dichlorobenzene	8270D	541-73-1	ug/L	NE	NE	2	1
1,4-Dichlorobenzene	8270D	106-46-7	ug/L	0.48	75	5	1
1,4-Dioxane	8270D	123-91-1	ug/L	0.46	NE	5	4
2,4,5-Trichlorophenol	8270D	95-95-4	ug/L	120	NE	2	1
2,4,6-Trichlorophenol	8270D	88-06-2	ug/L	1.2	NE	2	1
2,4-Dichlorophenol	8270D	120-83-2	ug/L	4.6	NE	2	1
2,4-Dimethylphenol	8270D	105-67-9	ug/L	36	NE	10	9
2,4-Dinitrophenol	8270D	51-28-5	ug/L	3.9	NE	30	28
2,4-Dinitrotoluene	8270D	121-14-2	ug/L	0.24	NE	5	2
2,6-Dinitrotoluene	8270D	606-20-2	ug/L	0.049	NE	2	1
2-Chloronaphthalene	8270D	91-58-7	ug/L	75	NE	1	0.8
2-Chlorophenol	8270D	95-57-8	ug/L	9.1	NE	2	1
2-Methylnaphthalene	8270D	91-57-6	ug/L	3.6	NE	0.5	0.2
2-Methylphenol	8270D	95-48-7	ug/L	93	NE	2	1
2-Nitroaniline	8270D	88-74-4	ug/L	19	NE	5	2
2-Nitrophenol	8270D	88-75-5	ug/L	NE	NE	5	2
3,3-Dichlorobenzidine	8270D	91-94-1	ug/L	0.13	NE	10	8
3,4-Methylphenol	8270D	108394/106445	ug/L	NE	NE	2	1
3-Nitroaniline	8270D	99-09-2	ug/L	NE	NE	5	4
4,6-Dinitro-2-methylphenol	8270D	534-52-1	ug/L	0.15	NE	21	20
4-Bromophenyl-phenylether	8270D	101-55-3	ug/L	NE	NE	2	1
4-Chloro-3-methylphenol	8270D	59-50-7	ug/L	140	NE	5	2
4-Chloroaniline	8270D	106-47-8	ug/L	0.37	NE	10	9
4-Chlorophenyl-phenylether	8270D	7005-72-3	ug/L	NE	NE	2	1
4-Nitroaniline	8270D	100-01-6	ug/L	3.8	NE	3	2
4-Nitrophenol	8270D	100-02-7	ug/L	NE	NE	30	20
Aniline	8270D	62-53-3	ug/L	13	NE	5	2
Benzyl Alcohol	8270D	100-51-6	ug/L	200	NE	10	8
Bis(2-chloro-1-methylethyl) ether	8270D	108-60-1	ug/L	71	NE	2	1
Bis(2-chloroethoxy)methane	8270D	111-91-1	ug/L	5.9	NE	2	1
Bis(2-chloroethyl)ether	8270D	111-44-4	ug/L	0.014	NE	2	1
Bis(2-ethylhexyl)phthalate	8270D	117-81-7	ug/L	5.6	6	5	4
Carbazole	8270D	86-74-8	ug/L	NE	NE	2	1
Dibenzofuran	8270D	132-64-9	ug/L	0.79	NE	2	1
Diethyl phthalate	8270D	84-66-2	ug/L	1500	NE	5	4
Dimethyl phthalate	8270D	131-11-3	ug/L	NE	NE	5	4
Di-n-butyl phthalate	8270D	84-74-2	ug/L	90	NE	5	4
Di-n-octyl phthalate	8270D	117-84-0	ug/L	20	NE	11	10
Diphenylamine	8270D	122-39-4	ug/L	130	NE	3	2
Hexachlorobenzene	8270D	118-74-1	ug/L	0.0098	1	0.5	0.22
Hexachlorobutadiene	8270D	87-68-3	ug/L	0.14	NE	2	1
hexachlorocyclopentadiene	8270D	77-47-4	ug/L	0.041	50	11	10
Hexachloroethane	8270D	67-72-1	ug/L	0.33	NE	5	1
Isophorone	8270D	78-59-1	ug/L	78	NE	2	1
Nitrobenzene	8270D	98-95-3	ug/L	0.14	NE	2	1
n-Nitroso-di-n-propylamine	8270D	621-64-7	ug/L	0.011	NE	2	1
n-Nitrosodiphenylamine	8270D	86-30-6	ug/L	12	NE	2	1
Pentachlorophenol	8270D	87-86-5	ug/L	0.041	1	5	4
Phenol	8270D	108-95-2	ug/L	580	NE	2	1
Pyridine	8270D	110-86-1	ug/L	2	NE	5	4
PAHs							
Acenaphthene	8270D-SIM	83-32-9	ug/L	53	NE	0.05	0.03
Acenaphthylene	8270D-SIM	208-96-8	ug/L	NE	NE	0.05	0.03
Anthracene	8270D-SIM	120-12-7	ug/L	180	NE	0.05	0.03
Benzo(a)anthracene	8270D-SIM	56-55-3	ug/L	0.03	NE	0.05	0.03
Benzo(a)pyrene	8270D-SIM	50-32-8	ug/L	0.025	0.2	0.05	0.03
Benzo(b)fluoranthene	8270D-SIM	205-99-2	ug/L	0.25	NE	0.05	0.03
Benzo(g,h,i)perylene	8270D-SIM	191-24-2	ug/L	NE	NE	0.05	0.03
Benzo(k)fluoranthene	8270D-SIM	207-08-9	ug/L	2.5	NE	0.05	0.03
Chrysene	8270D-SIM	218-01-9	ug/L	25	NE	0.05	0.03
Dibenz(a,h)anthracene	8270D-SIM	53-70-3	ug/L	0.025	NE	0.05	0.04
Fluoranthene	8270D-SIM	206-44-0	ug/L	80	NE	0.05	0.03
Fluorene	8270D-SIM	86-73-7	ug/L	29	NE	0.05	0.03
Indeno(1,2,3-cd)pyrene	8270D-SIM	193-39-5	ug/L	0.25	NE	0.05	0.04
Naphthalene	8270D-SIM	91-20-3	ug/L	0.12	NE	0.07	0.06
Phenanthrene	8270D-SIM	85-01-8	ug/L	NE	NE	0.07	0.06
Pyrene	8270D-SIM	129-00-0	ug/L	12	NE	0.05	0.03
Metals							
Aluminum	6020B	7429-90-5	ug/L	2000	NE	35	30
Antimony	6020B	7440-36-0	ug/L	0.78	6	1	0.8
Arsenic	6020B	7440-38-2	ug/L	0.052	10	2	1.6
Barium	6020B	7440-39-3	ug/L	380	2000	2	1.6
Beryllium	6020B	7440-41-7	ug/L	2.5	4	0.5	0.25
Cadmium	6020B	7440-43-9	ug/L	NE	5	0.5	0.4
Calcium (Ca)	6020B	7440-70-2	ug/L	NE	NE	125	120
Chromium	6020B	7440-47-3	ug/L	NE	100	2	0.8
Cobalt	6020B	7440-48-4	ug/L	0.6	NE	0.5	0.4

Table 4-5. SAP Reference Limits for Groundwater

Chemical	Method	CAS #	Unit	Screening Benchmarks		Laboratory Limits	
				Tap Water RSLs (2021, HQ=0.1)	Federal MCLs	LOQ	LOD
Copper	6020B	7440-50-8	ug/L	80	1300	1	0.8
Iron (Fe)	6020B	7439-89-6	ug/L	1400	NE	50	40
Lead	6020B	7439-92-1	ug/L	15	15	0.5	0.25
Magnesium (Mg)	6020B	7439-95-4	ug/L	NE	NE	50	25
Manganese (Mn)	6020B	7439-96-5	ug/L	NE	NE	2	1.6
Nickel	6020B	7440-02-0	ug/L	39	NE	1.5	1
Potassium (K)	6020B	7440-09-7	ug/L	NE	NE	200	160
Selenium	6020B	7782-49-2	ug/L	10	50	1	0.8
Silver	6020B	7440-22-4	ug/L	9.4	NE	0.5	0.4
Sodium (Na)	6020B	7440-23-5	ug/L	NE	NE	200	160
Thallium	6020B	7440-28-0	ug/L	0.02	2	0.5	0.4
Vanadium	6020B	7440-62-2	ug/L	8.6	NE	4	1.6
Zinc	6020B	7440-66-6	ug/L	600	NE	15	10
Mercury	7470A	7439-97-6	ug/L	0.063	2	0.2	0.16
Hexavalent Chromium	218.6	18540-29-9	ug/L	0.035	NE	10	9
Dioxins/Furans							
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	8290A	67562-39-4	ug/L	NE	NE	0.000025	0.00001
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	8290A	35822-46-9	ug/L	NE	NE	0.000025	0.00001
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	8290A	55673-89-7	ug/L	NE	NE	0.000025	0.00001
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	70648-26-9	ug/L	NE	NE	0.000025	0.00001
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	39227-28-6	ug/L	NE	NE	0.000025	0.00001
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	57117-44-9	ug/L	NE	NE	0.000025	0.00001
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	57653-85-7	ug/L	NE	NE	0.000025	0.00001
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	8290A	72918-21-9	ug/L	NE	NE	0.000025	0.00001
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	19408-74-3	ug/L	NE	NE	0.000025	0.00001
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	8290A	57117-41-6	ug/L	NE	NE	0.000025	0.00001
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	8290A	40321-76-4	ug/L	NE	NE	0.000025	0.00001
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	60851-34-5	ug/L	NE	NE	0.000025	0.00001
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	8290A	57117-31-4	ug/L	NE	NE	0.000025	0.00001
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	8290A	51207-31-9	ug/L	NE	NE	0.000005	0.000002
Octachlorodibenzofuran (OCDF)	8290A	39001-02-0	ug/L	NE	NE	0.00005	0.00002
Octachlorodibenzo-p-dioxin (OCDD)	8290A	3268-87-9	ug/L	NE	NE	0.00011	0.0000725
TCDD, 2,3,7,8-	8290A	1746-01-6	ug/L	0.00000012	0.00003	0.000005	0.000002
Radionuclides							
Gross alpha	900	12587-46-1	pCi/L	NE	15	3	NE
Gross beta	900	12587-47-2	pCi/L	NE	50	4	NE
Radium 226	903	13982-63-3	pCi/L	NE	5(1)	1	NE
Radium 228	904	15262-20-1	pCi/L	NE	5(1)	1	NE
Total Uranium	200.8	7440-61-1	ug/L	0.4	30	1	0.8

Denotes a laboratory limit that exceeds one or more project screening benchmarks.

(1) The MCL of 5 pCi/L is for the sum of Radium-226 and Radium-228.

NE - Not Established

Table 4-6. SAP Reference Limits for Sediment

Chemical	Method	CAS #	Unit	Screening Benchmarks				Laboratory Limits	
				EPA Res RSL (2021, HQ=0.1)	EPA Ind RSL (2021, HQ=0.1)	Freshwater Invertebrate ESV	Marine Invertebrate ESV	LOQ	LOD
Pesticides									
4,4'-DDD	8081B	72-54-8	mg/kg	0.19	2.5	0.00488	0.00122	0.0017	0.0012
4,4'-DDE	8081B	72-55-9	mg/kg	2	9.3	0.00316	0.002	0.0017	0.0012
4,4'-DDT	8081B	50-29-3	mg/kg	1.9	8.5	0.00416	0.001	0.0017	0.0016
Aldrin	8081B	309-00-2	mg/kg	0.039	0.18	7.4	3.19	0.00083	0.0006
alpha Chlordane	8081B	5103-71-9	mg/kg	3.6	50	NE	NE	0.00083	0.0006
alpha-BHC	8081B	319-84-6	mg/kg	0.086	0.36	0.03	NE	0.00083	0.0006
beta-BHC	8081B	319-85-7	mg/kg	0.3	1.3	0.03	NE	0.001	0.0009
Chlordane	8081B	12789-03-6	mg/kg	1.7	7.7	0.00324	0.0005	0.017	0.008
delta-BHC	8081B	319-86-8	mg/kg	NE	NE	0.14	NE	0.001	0.0009
Dieldrin	8081B	60-57-1	mg/kg	0.034	0.14	0.0019	0.00002	0.0017	0.0012
Endosulfan I	8081B	959-98-8	mg/kg	NE	NE	NE	NE	0.00083	0.0006
Endosulfan II	8081B	33213-65-9	mg/kg	NE	NE	0.0000064	0.000004	0.0023	0.0022
Endosulfan sulfate	8081B	1031-07-8	mg/kg	38	490	0.0000064	0.000004	0.0017	0.0012
Endrin	8081B	72-20-8	mg/kg	1.9	25	0.0022	NE	0.0017	0.0012
Endrin aldehyde	8081B	7421-93-4	mg/kg	NE	NE	0.0044	0.00028	0.0017	0.0012
Endrin ketone	8081B	53494-70-5	mg/kg	NE	NE	NE	NE	0.002	0.0018
gamma-BHC (Lindane)	8081B	58-89-9	mg/kg	0.57	2.5	0.00237	0.00032	0.00083	0.0006
gamma-Chlordane	8081B	5103-74-2	mg/kg	NE	NE	NE	NE	0.00083	0.0006
Heptachlor	8081B	76-44-8	mg/kg	0.13	0.63	0.05	0.0508	0.00083	0.00062
Heptachlor epoxide	8081B	1024-57-3	mg/kg	0.07	0.33	0.00247	NE	0.00083	0.0006
Methoxychlor	8081B	72-43-5	mg/kg	32	410	0.02	0.0293	0.0067	0.0065
Toxaphene	8081B	8001-35-2	mg/kg	0.49	2.1	0.00051	0.0005	0.033	0.028
PCBs									
Aroclor 1016	8082A	12674-11-2	mg/kg	0.41	5.1	NE	NE	0.017	0.01
Aroclor 1221	8082A	11104-28-2	mg/kg	0.2	0.83	NE	NE	0.017	0.01
Aroclor 1232	8082A	11141-16-5	mg/kg	0.17	0.72	NE	NE	0.017	0.01
Aroclor 1242	8082A	53469-21-9	mg/kg	0.23	0.95	NE	NE	0.017	0.01
Aroclor 1248	8082A	12672-29-6	mg/kg	0.23	0.94	NE	NE	0.017	0.01
Aroclor 1254	8082A	11097-69-1	mg/kg	0.12	0.97	NE	NE	0.017	0.01
Aroclor 1260	8082A	11096-82-5	mg/kg	0.24	0.99	NE	NE	0.017	0.01
Aroclor 1262	8082A	37324-23-5	mg/kg	NE	NE	NE	NE	0.017	0.01
Aroclor 1268	8082A	11100-14-4	mg/kg	NE	NE	NE	NE	0.017	0.01
Herbicides									
2,4,5-T	8151A	93-76-5	mg/kg	63	820	NE	NE	0.0017	0.00164
2,4,5-TP (Silvex)	8151A	93-72-1	mg/kg	51	660	NE	NE	0.0017	0.0015
2,4-D	8151A	94-75-7	mg/kg	70	960	NE	NE	0.036	0.024
2,4-DB	8151A	94-82-6	mg/kg	190	2500	NE	NE	0.021	.0.02
Dalapon	8151A	75-99-0	mg/kg	190	2500	NE	NE	0.09	0.088
Dicamba	8151A	1918-00-9	mg/kg	190	2500	NE	NE	0.012	0.008
Dichlorprop	8151A	120-36-5	mg/kg	NE	NE	NE	NE	0.02	0.018
Dinoseb	8151A	88-85-7	mg/kg	6.3	82	NE	NE	0.024	0.018
MCPA (2-Methyl-4-chlorophenoxy acetic acid)	8151A	94-74-6	mg/kg	3.2	41	NE	NE	2.5	1.52
MCPP (2-(2-Methyl-4-chlorophenoxy) propanoic acid)	8151A	93-65-2	mg/kg	6.3	82	NE	NE	7.7	7.6
VOCs									
1,1,1-Trichloroethane	8260C	71-55-6	mg/kg	810	3600	0.01	NE	0.005	0.002
1,1,2,2-Tetrachloroethane	8260C	79-34-5	mg/kg	0.6	2.7	0.57	NE	0.005	0.001
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	8260C	76-13-1	mg/kg	670	2800	NE	NE	0.01	0.002
1,1,2-Trichloroethane	8260C	79-00-5	mg/kg	0.15	0.63	0.6	NE	0.005	0.002
1,1-Dichloroethane	8260C	75-34-3	mg/kg	3.6	16	0.01	NE	0.005	0.002
1,1-Dichloroethene	8260C	75-35-4	mg/kg	23	100	0.01	NE	0.005	0.002
1,2,4-Trichlorobenzene	8260C	120-82-1	mg/kg	5.8	26	0.0961	0.0961	0.01	0.008
1,2-Dibromo-3-chloropropane	8260C	96-12-8	mg/kg	0.0053	0.064	NE	NE	0.005	0.001
1,2-Dibromoethane	8260C	106-93-4	mg/kg	0.036	0.16	NE	NE	0.005	0.001
1,2-Dichlorobenzene	8260C	95-50-1	mg/kg	180	930	NE	0.259	0.005	0.002
1,2-Dichloroethane	8260C	107-06-2	mg/kg	0.46	2	0.02	NE	0.005	0.002
1,2-Dichloropropane	8260C	78-87-5	mg/kg	1.6	6.6	NE	NE	0.005	0.002
1,3-Dichlorobenzene	8260C	541-73-1	mg/kg	NE	NE	0.44	NE	0.005	0.002
1,4-Dichlorobenzene	8260C	106-46-7	mg/kg	2.6	11	0.09	NE	0.005	0.001
2-Butanone	8260C	78-93-3	mg/kg	2700	19000	NE	NE	0.01	0.004
2-Hexanone	8260C	591-78-6	mg/kg	20	130	0.01	NE	0.01	0.004
4-Methyl-2-pentanone	8260C	108-10-1	mg/kg	3300	14000	0.03	NE	0.01	0.004
Acetone	8260C	67-64-1	mg/kg	6100	67000	0.27	NE	0.02	0.016
Benzene	8260C	71-43-2	mg/kg	1.2	5.1	0.08	0.0648	0.005	0.002
Bromodichloromethane	8260C	75-27-4	mg/kg	0.29	1.3	NE	NE	0.005	0.001
Bromoform	8260C	75-25-2	mg/kg	19	86	0.28	NE	0.01	0.008
Bromomethane	8260C	74-83-9	mg/kg	0.68	3	NE	NE	0.005	0.002
Carbon disulfide	8260C	75-15-0	mg/kg	77	350	NE	NE	0.005	0.002
Carbon tetrachloride	8260C	56-23-5	mg/kg	0.65	2.9	0.02	NE	0.005	0.002
Chlorobenzene	8260C	108-90-7	mg/kg	28	130	0.0028	0.0548	0.005	0.002
Chloroethane	8260C	75-00-3	mg/kg	NE	NE	NE	NE	0.005	0.004
Chloroform	8260C	67-66-3	mg/kg	0.32	1.4	0.00072	NE	0.005	0.002
Chloromethane	8260C	74-87-3	mg/kg	11	46	NE	NE	0.005	0.002
cis-1,2-Dichloroethene	8260C	156-59-2	mg/kg	16	230	NE	NE	0.005	0.002
cis-1,3-Dichloropropene	8260C	10061-01-5	mg/kg	NE	NE	NE	NE	0.005	0.001
cyclohexane	8260C	110-82-7	mg/kg	650	2700	NE	NE	0.005	0.002
Cyclohexanone	8260C	108-94-1	mg/kg	2800	13000	NE	NE	12.5	5
Dibromochloromethane	8260C	124-48-1	mg/kg	8.3	39	NE	NE	0.005	0.001
Dichlorodifluoromethane	8260C	75-71-8	mg/kg	8.7	37	NE	NE	0.005	0.002
Ethylbenzene	8260C	100-41-4	mg/kg	5.8	25	0.026	0.0908	0.005	0.001
Isopropylbenzene	8260C	98-82-8	mg/kg	190	990	NE	NE	0.005	0.001
Methyl tert-butyl ether	8260C	1634-04-4	mg/kg	47	210	NE	NE	0.005	0.002
Methylacetate	8260C	79-20-9	mg/kg	7800	0.000012	NE	NE	0.005	0.004
methylcyclohexane	8260C	108-87-2	mg/kg	NE	NE	NE	NE	0.005	0.002
Methylene chloride	8260C	75-09-2	mg/kg	35	320	NE	NE	0.005	0.004
Styrene	8260C	100-42-5	mg/kg	600	3500	0.56	NE	0.005	0.001
Tetrachloroethene	8260C	127-18-4	mg/kg	8.1	39	0.078	NE	0.005	0.002

Table 4-6. SAP Reference Limits for Sediment

Chemical	Method	CAS #	Unit	Screening Benchmarks				Laboratory Limits	
				EPA Res RSL (2021, HQ=0.1)	EPA Ind RSL (2021, HQ=0.1)	Freshwater Invertebrate ESV	Marine Invertebrate ESV	LOQ	LOD
Toluene	8260C	108-88-3	mg/kg	490	4700	NE	0.391	0.005	0.002
trans-1,2-Dichloroethene	8260C	156-60-5	mg/kg	7	30	0.31	NE	0.005	0.002
trans-1,3-Dichloropropene	8260C	10061-02-6	mg/kg	NE	NE	NE	NE	0.005	0.001
Trichloroethene	8260C	79-01-6	mg/kg	0.41	1.9	0.035	NE	0.005	0.002
Trichlorofluoromethane	8260C	75-69-4	mg/kg	2300	35000	NE	NE	0.005	0.002
Vinyl chloride	8260C	75-01-4	mg/kg	0.059	1.7	NE	NE	0.005	0.002
Xylenes (total)	8260C	1330-20-7	mg/kg	58	250	226	NE	0.01	0.002
SVOCs									
1,2,4-Trichlorobenzene	8270D	120-82-1	mg/kg	5.8	26	0.0961	0.0961	0.0367	0.0333
1,2-Dichlorobenzene	8270D	95-50-1	mg/kg	180	930	NE	0.259	0.0367	0.0333
1,3-Dichlorobenzene	8270D	541-73-1	mg/kg	NE	NE	0.44	NE	0.0367	0.0333
1,4-Dichlorobenzene	8270D	106-46-7	mg/kg	2.6	11	0.09	NE	0.0367	0.0333
1,4-Dioxane	8270D	123-91-1	mg/kg	5.3	24	NE	NE	0.167	0.0667
2,4,5-Trichlorophenol	8270D	95-95-4	mg/kg	630	8200	0.29	NE	0.0367	0.0333
2,4,6-Trichlorophenol	8270D	88-06-2	mg/kg	6.3	82	NE	NE	0.0367	0.0333
2,4-Dichlorophenol	8270D	120-83-2	mg/kg	19	250	NE	NE	0.0433	0.04
2,4-Dimethylphenol	8270D	105-67-9	mg/kg	130	1600	NE	NE	0.0367	0.0333
2,4-Dinitrophenol	8270D	51-28-5	mg/kg	13	160	NE	NE	1	0.333
2,4-Dinitrotoluene	8270D	121-14-2	mg/kg	1.7	7.4	NE	NE	0.167	0.0667
2,6-Dinitrotoluene	8270D	606-20-2	mg/kg	0.36	1.5	NE	NE	0.0367	0.0333
2-Chloronaphthalene	8270D	91-58-7	mg/kg	480	6000	NE	NE	0.0333	0.0267
2-Chlorophenol	8270D	95-57-8	mg/kg	39	580	0.027	NE	0.0367	0.0333
2-Methylnaphthalene	8270D	91-57-6	mg/kg	24	300	4.47	0.0202	0.0167	0.00999
2-Methylphenol	8270D	95-48-7	mg/kg	320	4100	0.012	NE	0.05	0.04
2-Nitroaniline	8270D	88-74-4	mg/kg	63	800	NE	NE	0.05	0.0333
2-Nitrophenol	8270D	88-75-5	mg/kg	NE	NE	NE	NE	0.05	0.04
3,3-Dichlorobenzidine	8270D	91-94-1	mg/kg	1.2	5.1	NE	NE	0.167	0.0667
3-Nitroaniline	8270D	99-09-2	mg/kg	NE	NE	NE	NE	0.167	0.0667
4,6-Dinitro-2-methylphenol	8270D	534-52-1	mg/kg	0.51	6.6	NE	NE	0.5	0.333
4-Bromophenyl-phenylether	8270D	101-55-3	mg/kg	NE	NE	0.26	NE	0.0367	0.0333
4-Chloro-3-methylphenol	8270D	59-50-7	mg/kg	630	8200	NE	NE	0.05	0.04
4-Chloroaniline	8270D	106-47-8	mg/kg	2.7	11	NE	NE	0.167	0.0667
4-Chlorophenyl-phenylether	8270D	7005-72-3	mg/kg	NE	NE	NE	NE	0.0367	0.0333
4-Nitroaniline	8270D	100-01-6	mg/kg	25	110	NE	NE	0.167	0.0667
4-Nitrophenol	8270D	100-02-7	mg/kg	NE	NE	NE	NE	0.5	0.333
Aniline	8270D	62-53-3	mg/kg	44	400	NE	NE	0.167	0.0667
Benzyl Alcohol	8270D	100-51-6	mg/kg	630	8200	NE	NE	0.5	0.333
Bis(2-chloro-1-methylethyl) ether	8270D	108-60-1	mg/kg	310	4700	NE	NE	0.0433	0.04
Bis(2-chloroethoxy)methane	8270D	111-91-1	mg/kg	19	250	NE	NE	0.0367	0.0333
Bis(2-chloroethyl)ether	8270D	111-44-4	mg/kg	0.23	1	NE	NE	0.0367	0.0333
Bis(2-ethylhexyl)phthalate	8270D	117-81-7	mg/kg	39	160	453	0.182	0.167	0.133
CARBAZOLE	8270D	86-74-8	mg/kg	NE	NE	NE	NE	0.0367	0.0333
Dibenzofuran	8270D	132-64-9	mg/kg	7.8	120	0.3	NE	0.0367	0.0333
Diethyl phthalate	8270D	84-66-2	mg/kg	5100	66000	0.6	NE	0.167	0.133
Dimethyl phthalate	8270D	131-11-3	mg/kg	NE	NE	NE	NE	0.167	0.133
Di-n-butyl phthalate	8270D	84-74-2	mg/kg	630	8200	1.2	NE	0.167	0.133
Di-n-octyl phthalate	8270D	117-84-0	mg/kg	63	820	17	NE	0.167	0.133
Hexachlorobenzene	8270D	118-74-1	mg/kg	0.21	0.96	NE	NE	0.0167	0.0133
Hexachlorobutadiene	8270D	87-68-3	mg/kg	1.2	5.3	0.7	NE	0.05	0.04
hexachlorocyclopentadiene	8270D	77-47-4	mg/kg	0.18	0.75	NE	NE	0.5	0.333
Hexachloroethane	8270D	67-72-1	mg/kg	1.8	8	0.21	NE	0.167	0.0667
Isophorone	8270D	78-59-1	mg/kg	570	2400	NE	NE	0.0667	0.0333
Nitrobenzene	8270D	98-95-3	mg/kg	5.1	22	NE	NE	0.0367	0.0333
n-Nitroso-di-n-propylamine	8270D	621-64-7	mg/kg	0.078	0.33	NE	NE	0.0667	0.05
n-Nitrosodiphenylamine	8270D	86-30-6	mg/kg	110	470	0.52	NE	0.0367	0.0333
Pentachlorophenol	8270D	87-86-5	mg/kg	1	4	NE	NE	0.167	0.133
Phenol	8270D	108-95-2	mg/kg	1900	25000	0.0012	NE	0.0367	0.0333
Pyridine	8270D	110-86-1	mg/kg	7.8	120	NE	NE	0.167	0.133
PAHs									
Acenaphthene	8270D-SIM	83-32-9	mg/kg	360	4500	4.91	0.00671	0.00167	0.00133
Acenaphthylene	8270D-SIM	208-96-8	mg/kg	NE	NE	4.52	0.00587	0.00167	0.00133
Anthracene	8270D-SIM	120-12-7	mg/kg	1800	23000	NE	0.0469	0.00167	0.00133
Benzo(a)anthracene	8270D-SIM	56-55-3	mg/kg	1.1	21	0.015	0.0748	0.00167	0.00133
Benzo(a)pyrene	8270D-SIM	50-32-8	mg/kg	0.11	2.1	0.032	0.0888	0.00167	0.00133
Benzo(b)fluoranthene	8270D-SIM	205-99-2	mg/kg	1.1	21	9.79	9.79	0.00167	0.00133
Benzo(g,h,i)perylene	8270D-SIM	191-24-2	mg/kg	NE	NE	0.016	10.95	0.00167	0.00133
Benzo(k)fluoranthene	8270D-SIM	207-08-9	mg/kg	11	210	9.81	9.81	0.00167	0.00133
Chrysene	8270D-SIM	218-01-9	mg/kg	110	2100	0.026	0.108	0.00167	0.00133
Dibenz(a,h)anthracene	8270D-SIM	53-70-3	mg/kg	0.11	2.1	0.033	0.00622	0.00167	0.00133
Fluoranthene	8270D-SIM	206-44-0	mg/kg	240	3000	0.031	0.113	0.00167	0.00133
Fluorene	8270D-SIM	86-73-7	mg/kg	240	3000	0.01	0.0212	0.00167	0.00133
Indeno(1,2,3-cd)pyrene	8270D-SIM	193-39-5	mg/kg	1.1	21	0.017	11.15	0.00167	0.00133
Naphthalene	8270D-SIM	91-20-3	mg/kg	2	8.6	NE	0.0346	0.00333	0.00267
Phenanthrene	8270D-SIM	85-01-8	mg/kg	NE	NE	0.019	0.0867	0.00233	0.002
Pyrene	8270D-SIM	129-00-0	mg/kg	180	2300	0.044	0.153	0.00167	0.00133
Metals									
Aluminum	6020B	7429-90-5	mg/kg	7700	110000	NE	NE	10	8
Antimony	6020B	7440-36-0	mg/kg	3.1	47	NE	2	0.2	0.1
Arsenic	6020B	7440-38-2	mg/kg	0.68	3	9.79	7.24	0.2	0.16
Barium	6020B	7440-39-3	mg/kg	1500	22000	NE	NE	0.2	0.16
Beryllium	6020B	7440-41-7	mg/kg	16	230	NE	NE	0.5	0.025
Cadmium	6020B	7440-43-9	mg/kg	7.1	98	0.583	0.68	0.05	0.04
Calcium (Ca)	6020B	7440-70-2	mg/kg	NE	NE	NE	NE	25	16
Chromium	6020B	7440-47-3	mg/kg	NE	NE	36.2	52.3	0.4	0.16
Cobalt	6020B	7440-48-4	mg/kg	2.3	35	NE	NE	0.1	0.05
Copper	6020B	7440-50-8	mg/kg	310	4700	28	18.7	0.2	0.16

Table 4-6. SAP Reference Limits for Sediment

Chemical	Method	CAS #	Unit	Screening Benchmarks				Laboratory Limits	
				EPA Res RSL (2021, HQ=0.1)	EPA Ind RSL (2021, HQ=0.1)	Freshwater Invertebrate ESV	Marine Invertebrate ESV	LOQ	LOD
Iron (Fe)	6020B	7439-89-6	mg/kg	5500	82000	188400	NE	10	10
Lead	6020B	7439-92-1	mg/kg	400	800	35.8	30.2	0.1	0.05
Magnesium (Mg)	6020B	7439-95-4	mg/kg	NE	NE	NE	NE	5	5
Manganese (Mn)	6020B	7439-96-5	mg/kg	NE	NE	631	NE	0.2	0.16
Nickel	6020B	7440-02-0	mg/kg	150	2200	19.5	15.9	0.2	0.16
Potassium (K)	6020B	7440-09-7	mg/kg	NE	NE	NE	NE	20	16
Selenium	6020B	7782-49-2	mg/kg	39	580	NE	NE	0.2	0.1
Silver	6020B	7440-22-4	mg/kg	39	580	NE	0.73	0.05	0.04
Sodium (Na)	6020B	7440-23-5	mg/kg	NE	NE	NE	NE	25	20
Thallium	6020B	7440-28-0	mg/kg	0.078	1.2	NE	NE	0.05	0.04
Vanadium	6020B	7440-62-2	mg/kg	39	580	NE	NE	0.4	0.1
Zinc	6020B	7440-66-6	mg/kg	2300	35000	98	120	15	0.75
Mercury	7471B	7439-97-6	mg/kg	1.1	4.6	0.18	0.13	0.083	0.066
Hexavalent Chromium	7199	18540-29-9	mg/kg	0.3	6.3	NE	NE	0.4	0.3
Dioxins/Furans									
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	8290A	67562-39-4	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	8290A	35822-46-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	8290A	55673-89-7	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	70648-26-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	39227-28-6	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	57117-44-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	57653-85-7	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	8290A	72918-21-9	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	8290A	19408-74-3	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	8290A	57117-41-6	mg/kg	NE	NE	NE	NE	0.000005	0.000004
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	8290A	40321-76-4	mg/kg	NE	NE	NE	NE	0.000005	0.000004
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	8290A	60851-34-5	mg/kg	NE	NE	NE	NE	0.000005	0.000004
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	8290A	57117-31-4	mg/kg	NE	NE	NE	NE	0.000005	0.000004
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	8290A	51207-31-9	mg/kg	NE	NE	NE	NE	0.000001	0.0000008
Octachlorodibenzofuran (OCDF)	8290A	39001-02-0	mg/kg	NE	NE	NE	NE	0.00001	0.000008
Octachlorodibenzo-p-dioxin (OCDD)	8290A	3268-87-9	mg/kg	NE	NE	NE	NE	0.00001	0.000008
TCDD, 2,3,7,8-	8290A	1746-01-6	mg/kg	0.0000048	0.000022	NE	NE	0.000001	0.0000008
Radionuclides[1]									
Uranium-238 (via Th-234)	901.1	7440-61-1	pCi/g	0.00176[1]	14[2]	227	NE	1	NE
Radium-226 (via Bi-214)	901.1	14733-03-0	pCi/g	0.00182[1]	0.6[2][3]	79.9	NE	1	NE
Thorium-232 (via Ac-228)	901.1	14331-83-0	pCi/g	0.00174[1]	1.1[2]	1300	NE	1	NE
Thorium-228 (Isotopic Thorium)	HASL 300	14274-82-9	pCi/g	0.00706[1]	4.7[2]	590	NE	1	NE
Thorium-230 (Isotopic Thorium)	HASL 300	14269-63-7	pCi/g	0.00182[1]	1.8[2]	4130	NE	1	NE
Thorium-232 (Isotopic Thorium)	HASL 300	7440-29-1	pCi/g	0.00174[1]	1.1[2]	1300	NE	1	NE
Uranium-234 (Isotopic Uranium)	HASL 300	13966-29-5	pCi/g	0.00179[1]	1.3[2]	202	NE	1	NE
Uranium-235 (Isotopic Uranium)	HASL 300	15117-96-1	pCi/g	0.00623[1]	8[2]	218	NE	1	NE
Uranium-238 (Isotopic Uranium)	HASL 300	7440-61-1	pCi/g	0.00176[1]	14[2]	227	NE	1	NE
Gross Alpha	900	12587-46-1	pCi/g	NE	NE	NE	NE	10	NE
Gross Beta	900	12587-47-2	pCi/g	NE	NE	NE	NE	10	NE

Denotes a laboratory limit that exceeds one or more project screening benchmarks.

NE - Not Established

(1) Residential Preliminary Remediation Goals for Radionuclides calculated using the USEPA PRG calculator <https://epa-prgs.ornl.gov/radionuclides/>.

(2) Human health soil screening benchmarks for radionuclides are based on soil screening levels for evaluating surface soil, as specified in US Nuclear Regulatory Commission Regulation (NUREG) 1757, Volume 1 (U.S. NRC, 2006).

(3) Screening level is based on Radium-226 in equilibrium with all its progeny.

Table 4-7. SAP Reference Limits for Soil Gas

Chemical	Method	CAS #	Unit	Screening Benchmarks	Laboratory Limits	
				USEPA Vapor Intrusion Screening Level[(1)]	RL	MDL
VOCs						
1,1,1-Trichloroethane	TO-15	71-55-6	ug/m^3	17400	2.7	0.68
1,1,2,2-Tetrachloroethane	TO-15	79-34-5	ug/m^3	1.61	3.4	0.89
1,1,2-Trichloroethane	TO-15	79-00-5	ug/m^3	0.695	2.7	0.59
1,1-Dichloroethane	TO-15	75-34-3	ug/m^3	58.5	2	0.68
1,1-Dichloroethene	TO-15	75-35-4	ug/m^3	695	2	1.06
1,2,4-Trichlorobenzene	TO-15	120-82-1	ug/m^3	6.95	15	5.89
1,2,4-Trimethylbenzene	TO-15	95-63-6	ug/m^3	209	2.4	1.06
1,2-Dibromoethane (EDB)	TO-15	106-93-4	ug/m^3	0.156	3.8	0.82
1,2-Dichlorobenzene	TO-15	95-50-1	ug/m^3	695	3	0.72
1,2-Dichloroethane	TO-15	107-06-2	ug/m^3	3.6	2	0.68
1,2-Dichloropropane	TO-15	78-87-5	ug/m^3	13.9	2.3	0.89
1,3,5-Trimethylbenzene	TO-15	108-67-8	ug/m^3	209	2.4	0.79
1,3-Butadiene	TO-15	106-99-0	ug/m^3	3.12	1.1	0.89
1,3-Dichlorobenzene	TO-15	541-73-1	ug/m^3	NE	3	0.84
1,4-Dichlorobenzene	TO-15	106-46-7	ug/m^3	8.51	3	0.73
1,4-Dioxane	TO-15	123-91-1	ug/m^3	18.7	7.2	0.79
2,2,4-Trimethylpentane	TO-15	540-84-1	ug/m^3	NE	2.3	0.56
2-Butanone (Methyl Ethyl Ketone)	TO-15	78-93-3	ug/m^3	17400	5.9	1.92
2-Hexanone	TO-15	591-78-6	ug/m^3	104	8.2	3.89
2-Propanol	TO-15	67-63-0	ug/m^3	695	4.9	1.68
3-Chloropropene	TO-15	107-05-1	ug/m^3	3.48	6.3	1.41
4-Ethyltoluene	TO-15	622-96-8	ug/m^3	NE	2.4	0.82
4-Methyl-2-pentanone	TO-15	108-10-1	ug/m^3	10400	2	0.61
Acetone	TO-15	67-64-1	ug/m^3	107000	12	3.90
alpha-Chlorotoluene	TO-15	100-44-7	ug/m^3	1.91	2.6	0.66
Benzene	TO-15	71-43-2	ug/m^3	12	1.6	0.56
Bromodichloromethane	TO-15	75-27-4	ug/m^3	2.53	3.4	0.95
Bromoform	TO-15	75-25-2	ug/m^3	85.1	5.2	0.94
Bromomethane	TO-15	74-83-9	ug/m^3	17.4	19	3.45
Carbon Disulfide	TO-15	75-15-0	ug/m^3	2430	6.2	2.25
Carbon Tetrachloride	TO-15	56-23-5	ug/m^3	15.6	3.1	0.62
Chlorobenzene	TO-15	108-90-7	ug/m^3	174	2.3	0.35
Chloroethane	TO-15	75-00-3	ug/m^3	34800	5.3	1.80
Chloroform	TO-15	67-66-3	ug/m^3	4.07	2.4	0.92
Chloromethane	TO-15	74-87-3	ug/m^3	313	10	2.25
cis-1,2-Dichloroethene	TO-15	156-59-2	ug/m^3	NE	2	0.84
cis-1,3-Dichloropropene	TO-15	10061-01-5	ug/m^3	NE	2.3	0.70
Cumene	TO-15	98-82-8	ug/m^3	1390	2.4	0.89
Cyclohexane	TO-15	110-82-7	ug/m^3	20900	1.7	0.30
Dibromochloromethane	TO-15	124-48-1	ug/m^3	NE	4.2	0.61
Ethanol	TO-15	64-17-5	ug/m^3	NE	3.8	2.98
Ethyl Benzene	TO-15	100-41-4	ug/m^3	37.4	2.2	0.60
Freon 11	TO-15	75-69-4	ug/m^3	NE	2.8	0.82
Freon 113	TO-15	76-13-1	ug/m^3	17400	3.8	0.87
Freon 114	TO-15	76-14-2	ug/m^3	17400	3.5	1.22
Freon 12	TO-15	75-71-8	ug/m^3	348	2.5	0.97
Heptane	TO-15	142-82-5	ug/m^3	1390	2	0.87
Hexachlorobutadiene	TO-15	87-68-3	ug/m^3	4.25	21	6.69
Hexane	TO-15	110-54-3	ug/m^3	2430	1.8	0.49
m,p-Xylene	TO-15	108-38-3	ug/m^3	348	2.2	0.63
Methyl tert-butyl ether	TO-15	1634-04-4	ug/m^3	360	7.2	0.93
Methylene Chloride	TO-15	75-09-2	ug/m^3	2090	17	2.62
Naphthalene (by request)	TO-15	91-20-3	ug/m^3	2.75	5.24	0.66
o-Xylene	TO-15	95-47-6	ug/m^3	348	2.2	0.67
Propylbenzene	TO-15	103-65-1	ug/m^3	3480	2.4	0.61
Styrene	TO-15	100-42-5	ug/m^3	3480	2.1	0.78
Tetrachloroethene	TO-15	127-18-4	ug/m^3	139	3.4	0.86
Tetrahydrofuran	TO-15	109-99-9	ug/m^3	6950	1.5	0.62
Toluene	TO-15	108-88-3	ug/m^3	17400	1.9	0.34
trans-1,2-Dichloroethene	TO-15	156-60-5	ug/m^3	139	2	0.95
trans-1,3-Dichloropropene	TO-15	10061-02-6	ug/m^3	NE	2.3	0.55
Trichloroethene	TO-15	79-01-6	ug/m^3	6.95	2.7	1.17
Vinyl Chloride	TO-15	75-01-4	ug/m^3	5.59	1.3	0.92

Denotes a laboratory limit that exceeds one or more project screening benchmarks.

NE - Not Established

RL = Reporting Limit

MDL = Method Detection Limit

(1) USEPA Residential VISLs (i.e., target sub-slab and near-source soil gas concentration) correspond to a to a target cancer risk of 1 x 10-6 and a hazard quotient of 0.1 and were retrieved from the USEPA VISL Calculator on October 22, 2021.

Table 4-8. Data Quality Indicators for Soil/Sediment

Data Quality Indicators (DQIs)	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
VOCs/SVOCs					
Accuracy/Bias	Soil/Sediment	VOCs/SVOCs	±30% recovery	Second source calibration verification.	A
Inertness of the injection port	Soil/Sediment	VOCs/SVOCs	≤20% for DDT	Breakdown check (SVOC analysis only)	A
Accuracy/Bias	Soil/Sediment	VOCs/SVOCs	As per DoD QSM V5.3	MS/MSD	S&A
Accuracy/Bias	Soil/Sediment	VOCs/SVOCs	Area count within -50 to +100% RT must be ± 30 seconds from the last calibration check	Internal Standards	A
Accuracy/Bias	Soil/Sediment	VOCs/SVOCs	As per DoD QSM V5.3	LCS	A
Accuracy/Bias	Soil/Sediment	VOCs/SVOCs	As per DoD QSM V5.3	Surrogate Spikes	A
Precision	Soil/Sediment	VOCs/SVOCs	RPD ≤30% when VOC/SVOC detects for both duplicates are ≥LOQ.	Laboratory Duplicates	A
Precision	Soil/Sediment	VOCs/SVOCs	RPD ≤30% when VOC/SVOC detects for both duplicates are ≥LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Soil/Sediment	VOCs/SVOCs	No target analytes detected > ½ LOQ	Equipment Blanks, Method Blanks, Trip Blanks (VOC only)	S&A
Sensitivity	Soil/Sediment	VOCs/SVOCs	±20% recovery at LOQ	Laboratory Fortified Blank at LOQ	A
Completeness	Soil/Sediment	VOCs/SVOCs	≥95%	Data Completeness Check	S&A
Metals					
Accuracy/Bias	Soil/Sediment	Metals	±10% recovery	Second source calibration verification.	A
Accuracy/Bias	Soil/Sediment	Metals	As per DoD QSM V5.3	MS/MSD	S&A
Accuracy/Bias	Soil/Sediment	Metals	±20% recovery	Interference check sample	A
Accuracy/Bias	Soil/Sediment	Metals	As per DoD QSM V5.3	LCS	A
Precision	Soil/Sediment	Metals	RPD ≤20% when detects for both duplicates are 5x LOQ.	Laboratory Duplicates	A
Precision	Soil/Sediment	Metals	RPD ≤20% when detects for both duplicates are 5X LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Soil/Sediment	Metals	No target analytes detected > ½LOQ	Equipment Blanks	S&A

Table 4-8. Data Quality Indicators for Soil/Sediment

Data Quality Indicators (DQIs)	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Accuracy/Bias Contamination	Soil/Sediment	Metals	No target analytes detected > LOD	Method Blanks	A
Sensitivity	Soil/Sediment	Metals	±20% recovery at LOQ	Laboratory Fortified Blank at LOQ	A
Completeness	Soil/Sediment	Metals	≥95%	Data Completeness Check	S&A
Herbicides/Pesticides/PCBs					
Accuracy/Bias	Soil/Sediment	Herbicides/Pesticides/PCBs	±20% of expected value from the ICAL	Second source calibration verification.	A
Inertness of the injection port	Soil/Sediment	Herbicides/Pesticides/PCBs	≤15% for both DDT and Endrin	Breakdown check (pesticide analysis only)	A
Accuracy/Bias	Soil/Sediment	Herbicides/Pesticides/PCBs	As per DoD QSM V5.3	MS/MSD	S&A
Accuracy/Bias	Soil/Sediment	Herbicides/Pesticides/PCBs	As per DoD QSM V5.3	LCS	A
Accuracy/Bias	Soil/Sediment	Herbicides/Pesticides/PCBs	As per DoD QSM V5.3	Surrogate Spikes	A
Precision	Soil/Sediment	Herbicides/Pesticides/PCBs	RPD ≤30% when Pesticides/PBCs/Herbicides detects for both duplicates are > LOQ.	Laboratory Duplicates	A
Precision	Soil/Sediment	Herbicides/Pesticides/PCBs	RPD ≤50% when Pesticides/PBCs/Herbicides detects for both duplicates are > LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Soil/Sediment	Herbicides/Pesticides/PCBs	No target analytes detected > ½ LOQ	Equipment Blanks, Method Blanks	S&A
Sensitivity	Soil/Sediment	Herbicides/Pesticides/PCBs	±40% recovery at LOQ	Laboratory Fortified Blank at LOQ	A
Completeness	Soil/Sediment	Herbicides/Pesticides/PCBs	≥95%	Data Completeness Check	S&A
Dioxins/Furans					
Accuracy/Bias	Soil/Sediment	Dioxins/Furans	All reported analytes and IS within ±20% of expected value	ICV and CCV	A
Accuracy/Bias	Soil/Sediment	Dioxins/Furans	As per DoD QSM V5.3	MS/MSD	S&A
Accuracy/Bias	Soil/Sediment	Dioxins/Furans	As per DoD QSM V5.3	LCS	A
Accuracy/Bias	Soil/Sediment	Dioxins/Furans	As per DoD QSM V5.3	Surrogate Spikes	A

Table 4-8. Data Quality Indicators for Soil/Sediment

Data Quality Indicators (DQIs)	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Precision	Soil/Sediment	Dioxins/Furans	RPD ≤20% when detects for both duplicates are > LOQ.	Laboratory Duplicates	A
Precision	Soil/Sediment	Dioxins/Furans	RPD ≤20% when detects for both duplicates are > LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Soil/Sediment	Dioxins/Furans	No target analytes detected > ½ LOQ	Equipment Blanks, Method Blanks	S&A
Completeness	Soil/Sediment	Dioxins/Furans	≥95%	Data Completeness Check	S&A
Radionuclides					
Precision	Soil/Sediment	Radionuclides	ND < 1.96	Field Duplicates	A
Precision	Soil/Sediment	Radionuclides	ND < 1.96	Laboratory Duplicates	A
Accuracy/Bias Contamination	Soil/Sediment	Radionuclides	Recovery (80-120%)	LCS and MS/MSD	A
Completeness	Soil/Sediment	Radionuclides	> 90%	Data Completeness Check	S&A

Table 4-9. Data Quality Indicators for Groundwater

Data Quality Indicators (DQIs)	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
VOCs and SVOCs					
Accuracy/Bias	Groundwater/Surface Water	VOCs/SVOCs	8270D - ±30% Recovery 8260C - ±30% Recovery	Second source calibration verification.	A
Inertness of the injection port	Groundwater/Surface Water	VOCs/SVOCs	≤20% for DDT	Breakdown check (SVOC analysis only)	A
Accuracy/Bias	Groundwater/Surface Water	VOCs/SVOCs	As per DoD Quality Systems Manual (QSM) V5.3	MS/MSD	S&A
Accuracy/Bias	Groundwater/Surface Water	VOCs/SVOCs	Area count within -50 to +100% RT must be +/- 30 seconds from the last calibration check	Internal Standards	A
Accuracy/Bias	Groundwater/Surface Water	VOCs/SVOCs	As per DoD QSM V5.3	LCS	A
Accuracy/Bias	Groundwater/Surface Water	VOCs/SVOCs	As per DoD QSM V5.3	Surrogate Spikes	A
Precision	Groundwater/Surface Water	VOCs/SVOCs	RPD ≤30% when VOC/SVOC detects for both duplicates are >LOQ.	Laboratory Duplicates	A
Precision	Groundwater/Surface Water	VOCs/SVOCs	RPD ≤30% when VOC/SVOC detects for both duplicates are ≥LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Groundwater/Surface Water	VOCs/SVOCs	No target analytes detected > ½ LOQ	Equipment Blanks, Method Blanks, Trip Blanks (VOC only)	S&A
Sensitivity	Groundwater/Surface Water	VOCs/SVOCs	±20% recovery at LOQ	Laboratory Fortified Blank at LOQ	A
Completeness	Groundwater/Surface Water	VOCs/SVOCs	≥95%	Data Completeness Check	S&A
Metals					
Accuracy/Bias	Groundwater/Surface Water	Metals	±10% recovery	Second source calibration verification	A
Accuracy/Bias	Groundwater/Surface Water	Metals	As per DoD QSM V5.3	MS/MSD	S&A
Accuracy/Bias	Groundwater/Surface Water	Metals	±20% recovery of true value	Interference check sample ISCAB	A
Accuracy/Bias	Groundwater/Surface Water	Metals	As per DoD QSM V5.3	LCS	A

Table 4-9. Data Quality Indicators for Groundwater

Data Quality Indicators (DQIs)	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Precision	Groundwater/Surface Water	Metals	RPD <20% when detects for both duplicates are 5x LOQ.	Laboratory Duplicates	A
Precision	Groundwater/Surface Water	Metals	RPD <20% when detects for both duplicates are 5x LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Groundwater/Surface Water	Metals	No target analytes detected > ½ LOQ	Equipment Blanks and Method Blanks	S&A
Accuracy/Bias Contamination	Groundwater/Surface Water	Metals	No target analytes detected > LOD	Calibration Blanks	A
Sensitivity	Groundwater/Surface Water	Metals	±20% recovery at LOQ	Laboratory Fortified Blank at LOQ	A
Completeness	Groundwater/Surface Water	Metals	≥95%	Data Completeness Check	S&A
Herbicides/Pesticides/PCBs					
Accuracy/Bias	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	±20% of expected value from the ICAL	Second source calibration verification	A
Inertness of the injection port	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	≤15% for both DDT and Endrin	Breakdown check (pesticide analysis only)	A
Accuracy/Bias	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	As per DoD QSM V5.3	MS/MSD	S&A
Accuracy/Bias	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	As per DoD QSM V5.3	LCS	A
Accuracy/Bias	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	As per DoD QSM V5.3	Surrogate Spikes	A
Precision	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	RPD ≤30% when Pesticides/PBCs/Herbicides detects for both duplicates are > LOQ.	Laboratory Duplicates	A
Precision	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	RPD ≤30% when Pesticides/PBCs/Herbicides detects for both duplicates are > LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	No target analytes detected > ½ LOQ	Equipment Blanks, Method Blanks	S&A
Sensitivity	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	±40% recovery at LOQ	Laboratory Fortified Blank at LOQ	A
Completeness	Groundwater/Surface Water	Herbicides/Pesticides/PCBs	≥95%	Data Completeness Check	S&A

Table 4-9. Data Quality Indicators for Groundwater

Data Quality Indicators (DQIs)	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Dioxins/Furans					
Accuracy/Bias	Groundwater/Surface Water	Dioxins/Furans	All reported analytes and IS within ± 20% of expected value	ICV and CCV	A
Accuracy/Bias	Groundwater/Surface Water	Dioxins/Furans	As per DoD QSM V5.3	MS/MSD	S&A
Accuracy/Bias	Groundwater/Surface Water	Dioxins/Furans	As per DoD QSM V5.3	LCS	A
Accuracy/Bias	Groundwater/Surface Water	Dioxins/Furans	As per DoD QSM V5.3	Surrogate Spikes	A
Precision	Groundwater/Surface Water	Dioxins/Furans	RPD ≤20% when detects for both duplicates are > LOQ.	Laboratory Duplicates	A
Precision	Groundwater/Surface Water	Dioxins/Furans	RPD ≤20% when detects for both duplicates are > LOQ.	Field Duplicates	S&A
Accuracy/Bias Contamination	Groundwater/Surface Water	Dioxins/Furans	No target analytes detected > ½ LOQ	Equipment Blanks, Method Blanks	S&A
Completeness	Groundwater/Surface Water	Dioxins/Furans	≥95%	Data Completeness Check	S&A
Radionuclides					
Accuracy/Bias	Groundwater/Surface Water	Radium 226/228	LCS ±3 σ of the mean.	LCS	A
Accuracy/Bias	Groundwater/Surface Water	Gross alpha/beta, Total Uranium	LCS ±3 σ of the mean.	LCS	S&A
Precision	Groundwater/Surface Water	Gross alpha/beta, Radium 226/228, Total Uranium	RPD < 30% when detects for both duplicates are >LOQ	Laboratory Duplicates	A
Precision	Groundwater/Surface Water	Gross alpha/beta, Radium 226/228, Total Uranium	RPD < 20% when detects for both duplicates are >LOQ	Field Duplicates	S&A
Accuracy/Bias Contamination	Groundwater/Surface Water	Gross alpha/beta, Radium 226/228, Total Uranium	No target analytes detected > ½ LOQ	Equipment Blanks, Method Blanks	S&A
Completeness	Groundwater/Surface Water	Gross alpha/beta, Radium 226/228, Total Uranium	≥95%	Data Completeness Check	S&A

Table 4-10. Data Quality Indicators for Soil Gas

Data Quality Indicators (DQIs)	Matrix	Parameter	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
VOCs					
Accuracy	Gas	VOCs	Laboratory generated QC limits	Surrogate Spike	A
Accuracy/Bias/Precision	Gas	VOCs	Recovery limits and RPDs per QSM 5.3 published limits Table C-43; RPD <30%1.	Laboratory Control Spike/Matrix Spike and their Duplicates	A
Accuracy/Laboratory Contamination	Gas	VOCs	No analytes detected >1/2 laboratory LOQ; Common lab contaminants must not be detected > LOQ.	Method Blank	A
Precision	Gas	VOCs	RPD ≤ 30%	Field Duplicate	S
Completeness	Gas	VOCs	≥95%	Data Completeness Check	S&A
Bias/Holding Time	Gas	VOCs	≤30 days preparation/analysis	Reported Sample Data	A
Sensitivity	Gas	VOCs	Laboratory Limits (RL, MDL)	Data validation	A

Table 4-11. Summary of Sample Handling Requirements

Analysis	Analytical Method	Container Type	No. Containers	Required Vol.	Preservation	Technical Holding Time
Soil/Sediment						
VOCs	SW-846 Method 8260C	40 ml vials, Terracore sampler	3 vials, 1 Terracore	15 g	Methanol, DI water, and/or Sodium Bisulfate; 2-6C	48 hours/14 days to analysis
SVOCs	SW-846 Method 8270D	8 oz. glass	1	30 g	none; 2-6C	14 days to prep/40 days to analysis
PAHs	SW-846 Method 8270D-SIM			30 g		14 days to prep/40 days to analysis
Inorganics	SW-846 Method 6020B			2 g		6 months
Mercury	SW-Method 7471B			2 g		28 days
Pesticides	SW-846 Method 8081B			30 g		14 days to prep/40 days to analysis
PCBs	SW-846 Method 8082A			30 g		14 days to prep/40 days to analysis
TOC	Lloyd-Kahn			10 g		14 days
Herbicides	SW-846 Method 8151A			30 g		14 days to prep/40 days to analysis
Dioxins/Furans	SW-846 Method 8290A	8 oz. amber glass	1	30 g	none; 2-6C	365 days to prep/365 days to analysis
Uranium-238, Radium-226 and Thorium-232	EPA 901.1m	32oz wide mouth poly	1	500 g	none	6 months
Isotopic Thorium and Uranium	HASL 300 / Alpha Spec (A-01-R)					
Gross Alpha and Gross Beta	EPA 900					
Groundwater						
VOCs	SW-846 Method 8260C	40 ml vial	3	120 ml	HCl; 2-6C	14 days
SVOCs	SW-846 Method 8270D	250 ml amber glass	4	250 ml	none; 2-6C	7 days to prep/40 days to analysis
PAHs	SW-846 Method 8270D-SIM			250 ml		7 days to prep/40 days to analysis
Pesticides	SW-846 Method 8081B			250 ml		7 days to prep/40 days to analysis
PCBs	SW-846 Method 8082A			250 ml		7 days to prep/40 days to analysis
Inorganics	SW-846 Method 6020B	250 ml plastic	1	50 ml	HNO_3; 2-6C	6 months
Mercury	SW-Method 7470A			2 ml		28 days
Dioxins/Furans	SW-846 Method 8290A	1000 ml amber glass	2	1000 ml	none; 2-6C	365 days to prep/365 days to analysis
Gross Alpha and Gross Beta	EPA 900	1L poly	3	200 ml	Nitric acid	6 months
Radium-226/228	EPA 904/903 Gas Flow Proportional			2 L		6 months
Total Uranium	EPA 200.8			100ml min		6 months
Herbicides	SW-846 Method 8151A	1L amber glass	2	1000 ml	none; 2-6C	7 days to prep/40 days to analysis
Soil Gas						
VOCs	SW-846 Method 8260C	1L Summa Canister	1	1 L	none, ambient temperature	30 days

Table 4-12. Field Equipment, Calibration, Maintenance, Testing, and Inspection

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person
Horiba U-52 Multi-Parameter Water Quality Sensor	Individual calibration of pH, specific conductivity, turbidity, dissolved oxygen, oxidation-reduction potential	As per manufacturer's instructions	NA	NA	Daily before use and post-use calibration check	Calibration accepted for each parameter	Re-calibrate if calibration error message appears in display or readings are unstable or inaccurate	Field Team leader
GEM^{TM}2000 Plus Gas Analyzer and Extraction Monitor	Calibration and field calibration using zero channels ambient (auto zero) and check span channels (calibration) with known calibration gas	As per manufactuer's instruction	As per manufacturer's instructions	Equipment inspected prior to use on-site and daily; Check Equip Calibration records.	Daily before use and post use calibration and if gas reading drift due to changes in ±20 degree change	Calibration accepted for each parameter.CO and H_2S ±10% Full Scale (FS)	Re-calibrate if calibration error message appears in display or readings are unstable or inaccurate	Field Team Leader
Geoprobe Rig	NA	As per manufacturer's instructions	As per manufacturer's instructions	Equipment Inspection Checklist	Daily before use	Successful inspection of applicable parts	Maintenance as per manufacturer's instructions	Drilling Rig Operator
Low Volume Air Samplers	Annual Calibration	N/A	Flow Calibration	Equipment inspected for damage prior to use on-Site; Calibration records reviewed by Project CHP/SRSL	Daily before use	Successful inspection of applicable parts	Maintenance as per manufacturer's instructions	Project CHP/SRSL
Bicron Microrem	Annual Calibration	Battery Check	Source Check			±20%	Repair/Recalibrate as necessary	
Ludlum Model 9 Ion Chamber	Annual Calibration	Battery Check	Source Check			±20%	Repair/Recalibrate as necessary	
Alpha/Beta Scintillation Detector (Ludlum 2360/43-93)	Annual Calibration	Battery Check	Source Check			±20%	Repair/Recalibrate as necessary	
Gamma Detector (Ludlum 2221/44-10-1) Waterproof	Annual Calibration	Battery Check	Source Check			±20%	Repair/Recalibrate as necessary	
Gamma Detector (Ludlum 2221/44-20)	Annual Calibration	Battery Check	Source Check			±20%	Repair/Recalibrate as necessary	
Geiger-Mueller Detector (Ludlum 3/44-9)	Annual Calibration	Battery Check	Source Check			±20%	Repair/Recalibrate as necessary	
Swipe/Air Sample Counter (Ludlum 3030E)	Annual Calibration	NA	Background/ Source Check			±3s	Repair/Recalibrate as necessary	
Downhole Gamma Detector (Ludlum 3/44-2)	Annual Calibration	Battery Check	Source Check			±20%	Repair/Recalibrate as necessary	
Heavy Equipment	NA	As per manufacturer's instructions	As per manufacturer's instructions	Equipment Inspection Checklist	Daily before use	Successful inspection of applicable parts	Contact rental company representative to perform maintenance as per manufacturer's instructions	Rental Company Representative

Table 4-13. Data Verification and Validation Process

<table>
<tr><th>Data Review Input</th><th>Description</th><th>Responsible for Verification</th><th>Step I/IIa/IIb</th></tr>
<tr><td>Holding Times</td><td>Review that the samples were shipped and stored at the required temperature. Ensure that the analyses were performed within the holding times. If holding times were not met, confirm that deviations were documented. Holding time examination will be documented in the data validation report.</td><td>Project Chemist</td><td>IIa</td></tr>
<tr><td>Sample results for representativeness</td><td>Check that the laboratory recorded both the temperature at sample receipt and the pH of the chemically preserved samples (if applicable) to ensure sample integrity was sustained from sample collection to analysis. Representativeness will be documented in the data validation report.</td><td>Project Chemist</td><td>IIa/IIb</td></tr>
<tr><td>Laboratory data results for accuracy</td><td>Ensure that the laboratory QC samples were analyzed and that the measurement performance criteria were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria were met. Accuracy will be documented in the data validation report.</td><td>Project Chemist</td><td>IIa/IIb</td></tr>
<tr><td>Field and laboratory duplicate analyses for precision</td><td>Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCS duplicates. Precision will be documented in the data validation report.</td><td>Project Chemist</td><td>IIa/IIb</td></tr>
<tr><td>Project action limits</td><td>Assess and document the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant on the other target compounds reported as undetected. Project action limit achievement will be documented in the data validation report.</td><td>Project Chemist</td><td>IIa/IIb</td></tr>
<tr><td>SAP QC sample documentation</td><td>Ensure that all QC samples were collected and analyzed and that the associated results were within acceptance limits. QC sample documentation will be documented in the data validation report</td><td>Project Chemist</td><td>IIa/IIb</td></tr>
<tr><td>Analytical data deviations</td><td>Determine the impact of any deviation from sampling or analytical methods, and laboratory SOP requirements and matrix interferences effect on the analytical results. Data deviations will be documented in the data validation report.</td><td>Project Chemist</td><td>IIb</td></tr>
<tr><td>Project quantitation limits for sensitivity</td><td>Ensure that the project LOD and LOQ were achieved. Project quantitation limit achievement will be documented in the data validation report.</td><td>Project Chemist</td><td>IIb</td></tr>
<tr><td>Validation</td><td>Validation qualifiers applied in accordance with National Functional Guidelines for organic and inorganic data review. Methods for which no data validation guidelines exist will be validated following the NFG deemed most appropriate by the data validator. Validation will be limited to reviewing laboratory quality control summary information and raw data will not be reviewed.</td><td>Project Chemist</td><td>IIa/IIb</td></tr>
<tr><td>Data qualifiers</td><td>Qualifiers that will be applied during the data validation process are summarized below and, as indicated, results will be considered usable unless qualified by an R-flag. Rejected data will be evaluated and may be used in circumstances identified by the Partnering Team.
<table>
<tr><th>Data Qualifier</th><th>Qualifier Definition</th><th>Interpret Result as Detection?</th><th>Result Usable?</th><th>Potential Result Bias</th></tr>
<tr><td>no qualifier</td><td>Acceptable</td><td>Yes</td><td>Yes</td><td>None expected</td></tr>
<tr><td>J+/J-</td><td>Estimated</td><td>Yes</td><td>Yes</td><td>High or Low</td></tr>
<tr><td>U</td><td>Undetected</td><td>No</td><td>Yes</td><td>None expected</td></tr>
<tr><td>UJ</td><td>Undetected and Estimated</td><td>No</td><td>Yes</td><td>High or Low</td></tr>
<tr><td>R</td><td>Rejected</td><td>No</td><td>No</td><td>Unspecified</td></tr>
</table></td><td>Project Chemist</td><td>IIa/IIb</td></tr>
</table>

Table 5-1. Sample Location Coordinates

Location ID	Latitude	Longitude
Site Surface and Subsurface Soil		
SCP-DPT-001	40.655033	-73.858095
SCP-DPT-002	40.654253	-73.857893
SCP-DPT-003	40.655187	-73.857070
SCP-DPT-004	40.654407	-73.856868
SCP-DPT-005	40.653626	-73.856666
SCP-DPT-006	40.652846	-73.856464
SCP-DPT-007	40.656121	-73.856248
SCP-DPT-008	40.655341	-73.856046
SCP-DPT-009	40.654561	-73.855843
SCP-DPT-010	40.653780	-73.855641
SCP-DPT-011	40.653000	-73.855439
SCP-DPT-012	40.652219	-73.855237
SCP-DPT-013	40.651439	-73.855035
SCP-DPT-014	40.657056	-73.855425
SCP-DPT-015	40.656275	-73.855223
SCP-DPT-016	40.655495	-73.855021
SCP-DPT-017	40.654715	-73.854819
SCP-DPT-018	40.653934	-73.854617
SCP-DPT-019	40.653154	-73.854414
SCP-DPT-020	40.652373	-73.854212
SCP-DPT-021	40.651593	-73.854010
SCP-DPT-022	40.650812	-73.853808
SCP-DPT-023	40.657210	-73.854400
SCP-DPT-024	40.656429	-73.854198
SCP-DPT-025	40.655649	-73.853996
SCP-DPT-026	40.654869	-73.853794
SCP-DPT-027	40.654088	-73.853592
SCP-DPT-028	40.653308	-73.853390
SCP-DPT-029	40.652527	-73.853188
SCP-DPT-030	40.651747	-73.852985
SCP-DPT-031	40.647064	-73.851773
SCP-DPT-032	40.646284	-73.851571
SCP-DPT-033	40.653462	-73.852365
SCP-DPT-034	40.652681	-73.852163
SCP-DPT-035	40.651901	-73.851961
SCP-DPT-036	40.647999	-73.850950
SCP-DPT-037	40.647218	-73.850748
SCP-DPT-038	40.646438	-73.850546
SCP-DPT-039	40.645657	-73.850344
SCP-DPT-040	40.653616	-73.851340
SCP-DPT-041	40.652835	-73.851138
SCP-DPT-042	40.652055	-73.850936
SCP-DPT-043	40.648933	-73.850128
SCP-DPT-044	40.648152	-73.849926
SCP-DPT-045	40.647372	-73.849724
SCP-DPT-046	40.646592	-73.849522
SCP-DPT-047	40.645811	-73.849320
SCP-DPT-048	40.645031	-73.849117
SCP-DPT-049	40.653769	-73.850316
SCP-DPT-050	40.652989	-73.850113
SCP-DPT-051	40.652209	-73.849911
SCP-DPT-052	40.650648	-73.849507
SCP-DPT-053	40.649867	-73.849305

Table 5-1. Sample Location Coordinates

Location ID	Latitude	Longitude
SCP-DPT-054	40.649087	-73.849103
SCP-DPT-055	40.648306	-73.848901
SCP-DPT-056	40.647526	-73.848699
SCP-DPT-057	40.646745	-73.848497
SCP-DPT-058	40.645965	-73.848295
SCP-DPT-059	40.645185	-73.848093
SCP-DPT-060	40.653923	-73.849291
SCP-DPT-061	40.653143	-73.849089
SCP-DPT-062	40.652362	-73.848887
SCP-DPT-063	40.651582	-73.848685
SCP-DPT-064	40.650802	-73.848483
SCP-DPT-065	40.650021	-73.848280
SCP-DPT-066	40.649241	-73.848078
SCP-DPT-067	40.648460	-73.847876
SCP-DPT-068	40.647680	-73.847674
SCP-DPT-069	40.646899	-73.847472
SCP-DPT-070	40.646119	-73.847270
SCP-DPT-071	40.645338	-73.847068
SCP-DPT-072	40.647053	-73.846448
SCP-DPT-073	40.646273	-73.846246
SCP-DPT-074	40.645492	-73.846044
SCP-DPT-075	40.644712	-73.845842
SCP-DPT-076	40.647207	-73.845423
SCP-DPT-077	40.646427	-73.845221
SCP-DPT-078	40.645646	-73.845019
SCP-DPT-079	40.644866	-73.844817
SCP-DPT-080	40.647361	-73.844398
SCP-DPT-081	40.646581	-73.844196
SCP-DPT-082	40.645800	-73.843994
SCP-DPT-083	40.647515	-73.843374
SCP-DPT-084	40.646734	-73.843172
SCP-DPT-085	40.645954	-73.842970
SCP-DPT-086	40.647669	-73.842349
SCP-DPT-087	40.646888	-73.842147
SCP-DPT-088	40.646108	-73.841945
SCP-DPT-089	40.645327	-73.841743
SCP-DPT-090	40.647823	-73.841324
SCP-DPT-091	40.647042	-73.841122
SCP-DPT-092	40.646262	-73.840921
SCP-DPT-093	40.645481	-73.840719
SCP-DPT-094	40.647976	-73.840300

Table 5-1. Sample Location Coordinates

Location ID	Latitude	Longitude
SCP-DPT-095	40.647196	-73.840098
SCP-DPT-096	40.646415	-73.839896
SCP-DPT-097	40.645635	-73.839694
SCP-DPT-098	40.648130	-73.839275
SCP-DPT-099	40.647350	-73.839073
SCP-DPT-100	40.646569	-73.838871
SCP-DPT-101	40.645789	-73.838669
SCP-DPT-102	40.648284	-73.838250
SCP-DPT-103	40.647503	-73.838049
SCP-DPT-104	40.646723	-73.837847
SCP-DPT-105	40.645943	-73.837645
SCP-DPT-106	40.648169	-73.847772
SCP-DPT-107	40.648603	-73.838386
Reference Surface and Subsurface Soil		
REF-DPT-001	40.656317	-73.860344
REF-DPT-002	40.656313	-73.859906
REF-DPT-003	40.656603	-73.860120
REF-DPT-004	40.656599	-73.859682
REF-DPT-005	40.656595	-73.859244
REF-DPT-006	40.656886	-73.859458
REF-DPT-007	40.656881	-73.859020
REF-DPT-008	40.656877	-73.858583
REF-DPT-009	40.657168	-73.858797
REF-DPT-010	40.657164	-73.858359
REF-DPT-011	40.657450	-73.858135
REF-DPT-012	40.657732	-73.857474
REF-DPT-013	40.658015	-73.856812
REF-DPT-014	40.658301	-73.856588
REF-DPT-015	40.658588	-73.856364
Site Groundwater		
SCP-MW-22	40.647834	-73.847977
SCP-MW-23	40.648475	-73.838244
Site Sediment		
SCP-SED-001	40.651258	-73.848866
SCP-SED-002	40.651116	-73.849252
SCP-SED-003	40.658000	-73.854547
SCP-SED-004	40.657695	-73.855269
SCP-SED-005	40.654725	-73.858729
Reference Sediment		
REF-SED-001	40.660193	-73.854105
REF-SED-002	40.660085	-73.854871
REF-SED-003	40.659704	-73.855296
REF-SED-004	40.659458	-73.855863
REF-SED-005	40.659087	-73.856359

Table 5-1. Sample Location Coordinates

Location ID	Latitude	Longitude
Site Soil Gas		
SCP-SG-001	40.657349	-73.854532
SCP-SG-002	40.655241	-73.854026
SCP-SG-003	40.653357	-73.852519
SCP-SG-004	40.653767	-73.849433
SCP-SG-005	40.652608	-73.848216
SCP-SG-006	40.650679	-73.847982
SCP-SG-007	40.648389	-73.847380
SCP-SG-008	40.647653	-73.844555
SCP-SG-009	40.648190	-73.841485
SCP-SG-010	40.648605	-73.838709

Table 5-2. Sample Analysis Summary for Surface Soil

Location ID	Sample ID	Field Parameters			Laboratory Analysis								
		PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
Site Systematic Grid-Based Surface Soil Samples[2]													
SCP-DPT-001	SCP-SS001	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-002	SCP-SS002	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-003	SCP-SS003	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-004	SCP-SS004	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-005	SCP-SS005	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-006	SCP-SS006	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-007	SCP-SS007	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-008	SCP-SS008	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-009	SCP-SS009	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-010	SCP-SS010	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-011	SCP-SS011	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-012	SCP-SS012	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-013	SCP-SS013	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-014	SCP-SS014	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-015	SCP-SS015	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-016	SCP-SS016	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-017	SCP-SS017	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-018	SCP-SS018	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-019	SCP-SS019	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-020	SCP-SS020	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-021	SCP-SS021	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-022	SCP-SS022	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-023	SCP-SS023	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-024	SCP-SS024	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-025	SCP-SS025	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-026	SCP-SS026	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-027	SCP-SS027	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-028	SCP-SS028	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-029	SCP-SS029	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-030	SCP-SS030	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-031	SCP-SS031	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-032	SCP-SS032	X	X	X	X	X	X	X	X	X	X	X	X

Table 5-2. Sample Analysis Summary for Surface Soil

		Field Parameters			Laboratory Analysis								
Location ID	Sample ID	PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
SCP-DPT-033	SCP-SS033	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-034	SCP-SS034	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-035	SCP-SS035	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-036	SCP-SS036	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-037	SCP-SS037	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-038	SCP-SS038	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-039	SCP-SS039	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-040	SCP-SS040	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-041	SCP-SS041	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-042	SCP-SS042	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-043	SCP-SS043	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-044	SCP-SS044	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-045	SCP-SS045	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-046	SCP-SS046	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-047	SCP-SS047	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-048	SCP-SS048	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-049	SCP-SS049	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-050	SCP-SS050	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-051	SCP-SS051	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-052	SCP-SS052	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-053	SCP-SS053	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-054	SCP-SS054	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-055	SCP-SS055	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-056	SCP-SS056	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-057	SCP-SS057	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-058	SCP-SS058	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-059	SCP-SS059	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-060	SCP-SS060	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-061	SCP-SS061	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-062	SCP-SS062	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-063	SCP-SS063	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-064	SCP-SS064	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-065	SCP-SS065	X	X	X	X	X	X	X	X	X	X	X	X

Table 5-2. Sample Analysis Summary for Surface Soil

Location ID	Sample ID	Field Parameters			Laboratory Analysis								
		PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
SCP-DPT-066	SCP-SS066	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-067	SCP-SS067	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-068	SCP-SS068	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-069	SCP-SS069	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-070	SCP-SS070	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-071	SCP-SS071	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-072	SCP-SS072	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-073	SCP-SS073	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-074	SCP-SS074	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-075	SCP-SS075	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-076	SCP-SS076	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-077	SCP-SS077	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-078	SCP-SS078	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-079	SCP-SS079	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-080	SCP-SS080	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-081	SCP-SS081	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-082	SCP-SS082	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-083	SCP-SS083	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-084	SCP-SS084	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-085	SCP-SS085	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-086	SCP-SS086	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-087	SCP-SS087	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-088	SCP-SS088	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-089	SCP-SS089	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-090	SCP-SS090	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-091	SCP-SS091	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-092	SCP-SS092	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-093	SCP-SS093	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-094	SCP-SS094	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-095	SCP-SS095	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-096	SCP-SS096	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-097	SCP-SS097	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-098	SCP-SS098	X	X	X	X	X	X	X	X	X	X	X	X

Table 5-2. Sample Analysis Summary for Surface Soil

Location ID	Sample ID	Field Parameters			Laboratory Analysis								
		PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
SCP-DPT-099	SCP-SS099	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-100	SCP-SS100	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-101	SCP-SS101	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-102	SCP-SS102	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-103	SCP-SS103	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-104	SCP-SS104	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-105	SCP-SS105	X	X	X	X	X	X	X	X	X	X	X	X
Site Biased Surface Soil Samples in the 1951 and 1954 Support Areas													
SCP-DPT-106	SCP-SS106	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-107	SCP-SS107	X	X	X	X	X	X	X	X	X	X	X	X
Site Surface Soil Samples Collected During Focused Radiological Investigations													
SCP-A1-1[3]	SCP-SS0A01-1	NA	NA	NA									X
Reference Surface Soil Samples[2]													
REF-DPT-001	REF-SS001	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-002	REF-SS002	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-003	REF-SS003	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-004	REF-SS004	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-005	REF-SS005	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-006	REF-SS006	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-007	REF-SS007	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-008	REF-SS008	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-009	REF-SS009	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-010	REF-SS010	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-011	REF-SS011	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-012	REF-SS012	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-013	REF-SS013	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-014	REF-SS014	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-015	REF-SS015	NA	NA	NA	X	X	X	X	X	X	X	X	X
Field QC Samples[4]													
Field Duplicates (10%)		NA	NA	NA	X	X	X	X	X	X	X	X	X
Equipment Blanks (10%)		NA	NA	NA	X	X	X	X	X	X	X	X	X
MS/MSDs (5%)		NA	NA	NA	X	X	X	X	X	X	X	X	X

Table 5-2. Sample Analysis Summary for Surface Soil

Location ID	Sample ID	Field Parameters			Laboratory Analysis								
		PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
Trip Blanks (Daily)		NA	NA	NA	X	NA	NA	NA	NA	NA	NA	NA	NA

Table 5-2. Sample Analysis Summary for Surface Soil

		Field Parameters			Laboratory Analysis								
Location ID	Sample ID	PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]

1 - Radiological analysis for soil includes uranium-238, radium-226 and thorium-232 via EPA Method 901.1, isotopic uranium and thorium via HASL 300, and gross alpha and gross beta via EPA 900.

2 - 25% of surface soil samples will be analyzed for TOC (Lloyd-Kahn Method).

3 - Soil samples collected during focused radiological investigations will be labeled according to an alpha-numeric grid cell in which the sample is collected. The alpha numeric-grid will be established following the gamma walkover surveys; the example above represents a sample collected at intrusive investigation #1 from Grid 0A01.

4 - QC samples will be collected to meet the specified sample frequency and the number of required QC samples for soil is dependent on the number of total samples collected. The sample labeling convention for field QC samples is discussed in Section 5.7; the sample IDs for field QC samples are dependent on the date on which they are collected.

NA- Not Applicable

Table 5-3. Sample Analysis Summary for Subsurface Soil

Location ID	Sample ID	Boring Target Depth	Field Parameters			Laboratory Analysis								
			PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
Site Systematic Grid-Based Subsurface Soil Samples[2]														
SCP-DPT-001	SCP-SU001	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-002	SCP-SU002	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-003	SCP-SU003	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-004	SCP-SU004	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-005	SCP-SU005	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-006	SCP-SU006	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-007	SCP-SU007	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-008	SCP-SU008	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-009	SCP-SU009	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-010	SCP-SU010	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-011	SCP-SU011	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-012	SCP-SU012	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-013	SCP-SU013	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-014	SCP-SU014	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-015	SCP-SU015	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-016	SCP-SU016	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-017	SCP-SU017	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-018	SCP-SU018	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-019	SCP-SU019	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-020	SCP-SU020	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-021	SCP-SU021	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-022	SCP-SU022	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-023	SCP-SU023	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-024	SCP-SU024	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-025	SCP-SU025	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-026	SCP-SU026	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-027	SCP-SU027	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-028	SCP-SU028	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-029	SCP-SU029	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-030	SCP-SU030	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-031	SCP-SU031	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-032	SCP-SU032	10 ft	X	X	X	X	X	X	X	X	X	X	X	X

Table 5-3. Sample Analysis Summary for Subsurface Soil

Location ID	Sample ID	Boring Target Depth	Field Parameters			Laboratory Analysis								
			PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
SCP-DPT-033	SCP-SU033	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-034	SCP-SU034	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-035	SCP-SU035	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-036	SCP-SU036	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-037	SCP-SU037	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-038	SCP-SU038	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-039	SCP-SU039	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-040	SCP-SU040	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-041	SCP-SU041	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-042	SCP-SU042	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-043	SCP-SU043	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-044	SCP-SU044	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-045	SCP-SU045	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-046	SCP-SU046	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-047	SCP-SU047	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-048	SCP-SU048	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-049	SCP-SU049	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-050	SCP-SU050	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-051	SCP-SU051	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-052	SCP-SU052	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-053	SCP-SU053	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-054	SCP-SU054	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-055	SCP-SU055	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-056	SCP-SU056	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-057	SCP-SU057	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-058	SCP-SU058	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-059	SCP-SU059	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-060	SCP-SU060	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-061	SCP-SU061	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-062	SCP-SU062	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-063	SCP-SU063	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-064	SCP-SU064	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-065	SCP-SU065	10 ft	X	X	X	X	X	X	X	X	X	X	X	X

Table 5-3. Sample Analysis Summary for Subsurface Soil

Location ID	Sample ID	Boring Target Depth	Field Parameters			Laboratory Analysis								
			PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
SCP-DPT-066	SCP-SU066	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-067	SCP-SU067	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-068	SCP-SU068	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-069	SCP-SU069	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-070	SCP-SU070	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-071	SCP-SU071	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-072	SCP-SU072	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-073	SCP-SU073	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-074	SCP-SU074	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-075	SCP-SU075	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-076	SCP-SU076	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-077	SCP-SU077	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-078	SCP-SU078	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-079	SCP-SU079	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-080	SCP-SU080	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-081	SCP-SU081	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-082	SCP-SU082	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-083	SCP-SU083	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-084	SCP-SU084	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-085	SCP-SU085	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-086	SCP-SU086	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-087	SCP-SU087	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-088	SCP-SU088	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-089	SCP-SU089	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-090	SCP-SU090	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-091	SCP-SU091	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-092	SCP-SU092	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-093	SCP-SU093	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-094	SCP-SU094	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-095	SCP-SU095	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-096	SCP-SU096	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-097	SCP-SU097	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-098	SCP-SU098	10 ft	X	X	X	X	X	X	X	X	X	X	X	X

Table 5-3. Sample Analysis Summary for Subsurface Soil

Location ID	Sample ID	Boring Target Depth	Field Parameters			Laboratory Analysis								
			PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
SCP-DPT-099	SCP-SU099	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-100	SCP-SU100	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-101	SCP-SU101	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-102	SCP-SU102	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-103	SCP-SU103	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-104	SCP-SU104	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-105	SCP-SU105	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
Site Biased Subsurface Soil Samples in the 1951 and 1954 Support Areas														
SCP-DPT-106	SCP-SU106	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
SCP-DPT-107	SCP-SU107	10 ft	X	X	X	X	X	X	X	X	X	X	X	X
Site Subsurface Soil Samples Collected During Focused Radiological Investigations														
SCP-A1-1[3]	SCP-SS0A01-1-5	NA	NA	NA	NA									X
Reference Subsurface Soil Samples														
REF-DPT-001	REF-SU001	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-002	REF-SU002	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-003	REF-SU003	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-004	REF-SU004	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-005	REF-SU005	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-006	REF-SU006	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-007	REF-SU007	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-008	REF-SU008	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-009	REF-SU009	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-010	REF-SU010	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-011	REF-SU011	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-012	REF-SU012	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-013	REF-SU013	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-014	REF-SU014	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
REF-DPT-015	REF-SU015	10 ft	NA	NA	NA	X	X	X	X	X	X	X	X	X
Field QC Samples[4]														
Field Duplicates (10%)		NA	NA	NA	NA	X	X	X	X	X	X	X	X	X
Equipment Blanks (10%)		NA	NA	NA	NA	X	X	X	X	X	X	X	X	X
MS/MSDs (5%)		NA	NA	NA	NA	X	X	X	X	X	X	X	X	X

Table 5-3. Sample Analysis Summary for Subsurface Soil

			Field Parameters			Laboratory Analysis								
Location ID	Sample ID	Boring Target Depth	PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
Trip Blanks (Daily)		NA	NA	NA	NA	X	NA	NA	NA	NA	NA	NA	NA	NA

Table 5-3. Sample Analysis Summary for Subsurface Soil

			Field Parameters			Laboratory Analysis								
Location ID	Sample ID	Boring Target Depth	PID	Downhole Gamma Scans	Soil Core Gamma Scans	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]

1 - Radiological analysis for soil includes uranium-238, radium-226 and thorium-232 via EPA Method 901.1, isotopic uranium and thorium via HASL 300, and gross alpha and gross beta via EPA 900.

2 - Up to two subsurface soil samples will be collected from the top 10 ft of the vadose zone and/or to the bottom of the waste fill layer. The sample ID for all subsurface samples will be expanded to specify the sample depth as a suffix of digit depth in feet bgs (e.g., SCP-SU001-3 for a soil sample collected at 3 ft bgs).

3 - Soil samples collected during focused radiological investigations will be labeled according to the alpha-numeric grid cell in which the sample is collected. The alpha numeric-grid will be established following the gamma walkover surveys; the example above represents a sample collected at intrusive investigation #1 from Grid 0A01 at a depth of 5 ft bgs.

4 - QC samples will be collected to meet the specified sample frequency and the number of required QC samples for soil is dependent on the number of total samples collected. The sample labeling convention for field QC samples is discussed in Section 5.7; the sample IDs for field QC samples are dependent on the date on which they are collected.

NA- Not Applicable

Table 5-4. Sample Analysis Summary for Groundwater

Location ID	Sample ID	Field Parameters						Laboratory Analysis								
		Temp	pH	Cond	DO	ORP	Turb	VOCs (8260C)	SVOCs (8270D)	PAHs (8270D-SIM)	Metals (6020B/ 7471B/ 218.6)	Herbicides (8151A)	Pesticides (8081B)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
Site Groundwater Samples																
MW-22	SCP-MW022	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
MW-23	SCP-MW023	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Field QC Samples[2]																
Field Duplicates (10%)		NA	NA	NA	NA	NA	NA	X	X	X	X	X	X	X	X	X
Equipment Blanks (10%)		NA	NA	NA	NA	NA	NA	X	X	X	X	X	X	X	X	X
MS/MSDs (5%)		NA	NA	NA	NA	NA	NA	X	X	X	X	X	X	X	X	X
Trip Blanks (Daily)		NA	NA	NA	NA	NA	NA	X	NA	NA	NA	NA	NA	NA	NA	NA

1 - Radiological analysis for groundwater includes gross alpha and gross beta via EPA Method 900, Radium-226/228 via EPA Method 904/903, and total Uranium via EPA Method 200.8.

2 - QC samples will be collected to meet the specified sample frequency. The sample labeling convention for field QC samples is discussed in Section 5.7; the sample IDs for field QC samples are dependent on the date on which they are collected.

NA- Not Applicable

Table 5-5. Sample Analysis Summary for Sediment

Location ID	Sample ID	Laboratory Analysis								
		VOCs (8260C)	SVOCs (8270D)	PAHs (8270D SIM)	Metals (6020B/ 7471B/ 7199)	Pesticides (8081B)	Herbicides (8151A)	PCBs (8082A)	Dioxins/ Furans (8290A)	Rad[1]
Site Sediment Samples[2]										
SCP-SE-001	SCP-SE001	X	X	X	X	X	X	X	X	X
SCP-SE-002	SCP-SE002	X	X	X	X	X	X	X	X	X
SCP-SE-003	SCP-SE003	X	X	X	X	X	X	X	X	X
SCP-SE-004	SCP-SE004	X	X	X	X	X	X	X	X	X
SCP-SE-005	SCP-SE005	X	X	X	X	X	X	X	X	X
Reference Sediment Samples[2]										
REF-SE-001	REF-SE001	X	X	X	X	X	X	X	X	X
REF-SE-002	REF-SE002	X	X	X	X	X	X	X	X	X
REF-SE-003	REF-SE003	X	X	X	X	X	X	X	X	X
REF-SE-004	REF-SE004	X	X	X	X	X	X	X	X	X
REF-SE-005	REF-SE005	X	X	X	X	X	X	X	X	X
Field QC Samples[3]										
Field Duplicates (10%)		X	X	X	X	X	X	X	X	X
Equipment Blanks (10%)		X	X	X	X	X	X	X	X	X
MS/MSDs (5%)		X	X	X	X	X	X	X	X	X
Trip Blanks (Daily)		X	NA	NA	NA	NA	NA	NA	NA	NA

1 - Radiological analysis for soil includes uranium-238, radium-226 and thorium-232 via EPA Method 901.1, isotopic uranium and thorium via HASL 300, and gross alpha and gross beta via EPA 900.

2 - 100% of sediment samples will be analyzed for TOC (Llyod-Kahn Method).

3 - QC samples will be collected to meet the specified sample frequency and the number of required QC samples for sediment is dependent on the number of total samples collected. The sample labeling convention for field QC samples is discussed in Section 5.7; the sample IDs for field QC samples are dependent on the date on which they are collected.

NA- Not Applicable

Table 5-6. Sample Analysis Summary for Soil Gas

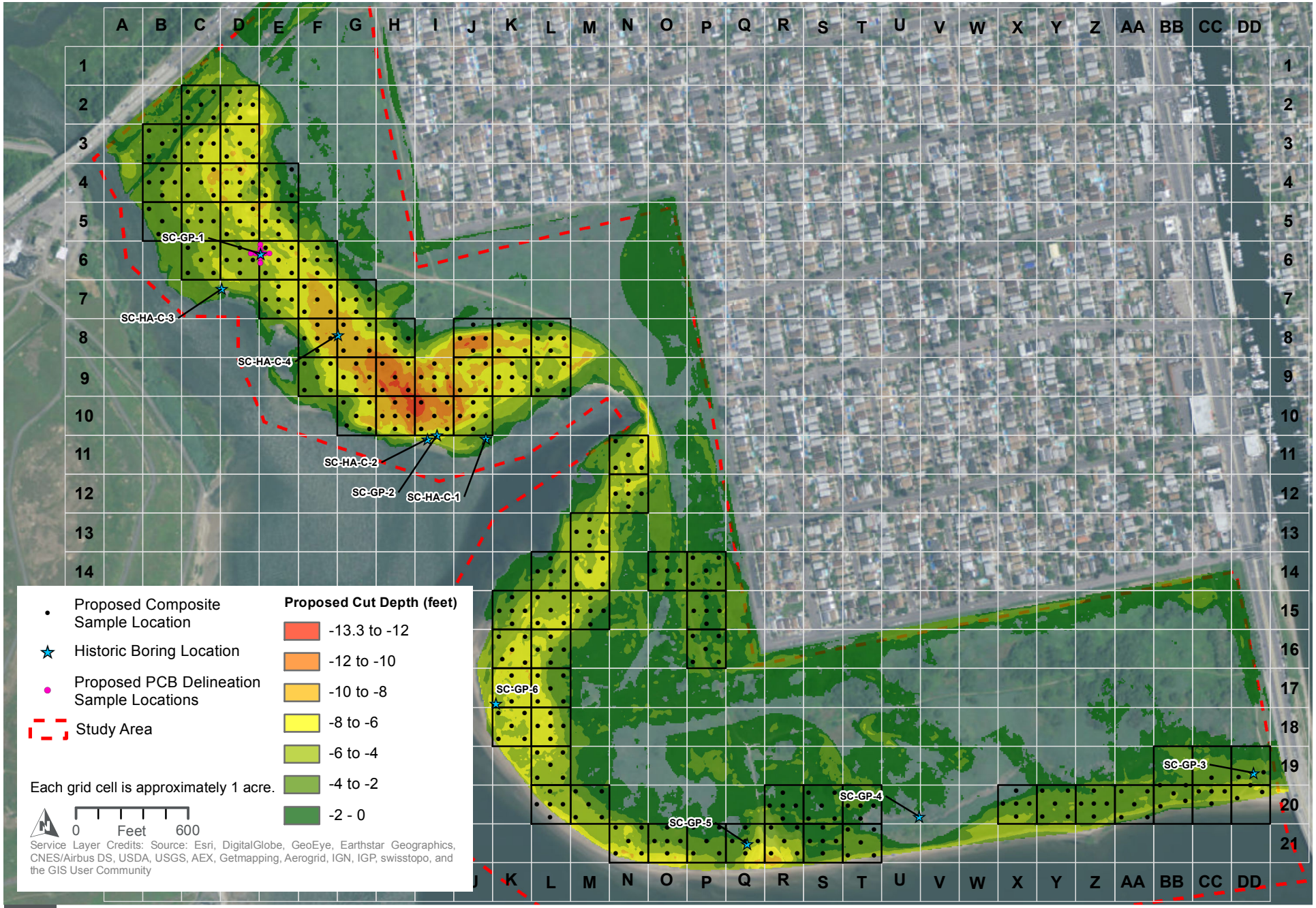
Location ID	Sample ID	Field Parameters						Laboratory Analysis
		CH_4	CO_2	O_2	CO	H_2S	Temp, Pressure, Gas Flow Rate	VOCs (TO-15)
Site Soil Gas Samples								
SCP-SG-001	SCP-SG001	X	X	X	X	X	X	X
SCP-SG-002	SCP-SG002	X	X	X	X	X	X	X
SCP-SG-003	SCP-SG003	X	X	X	X	X	X	X
SCP-SG-004	SCP-SG004	X	X	X	X	X	X	X
SCP-SG-005	SCP-SG005	X	X	X	X	X	X	X
SCP-SG-006	SCP-SG006	X	X	X	X	X	X	X
SCP-SG-007	SCP-SG007	X	X	X	X	X	X	X
SCP-SG-008	SCP-SG008	X	X	X	X	X	X	X
SCP-SG-009	SCP-SG009	X	X	X	X	X	X	X
SCP-SG-010	SCP-SG010	X	X	X	X	X	X	X
Field QC Samples[1]								
Field Duplicates (10%)		NA	NA	NA	NA	NA	NA	X

1 - QC samples will be collected to meet the specified sample frequency. The sample labeling convention for field QC samples is discussed in Section 5.7; the sample IDs for field QC samples are dependent on the date on which they are collected.

NA- Not Applicable

Appendix A – Previous Investigation Figures

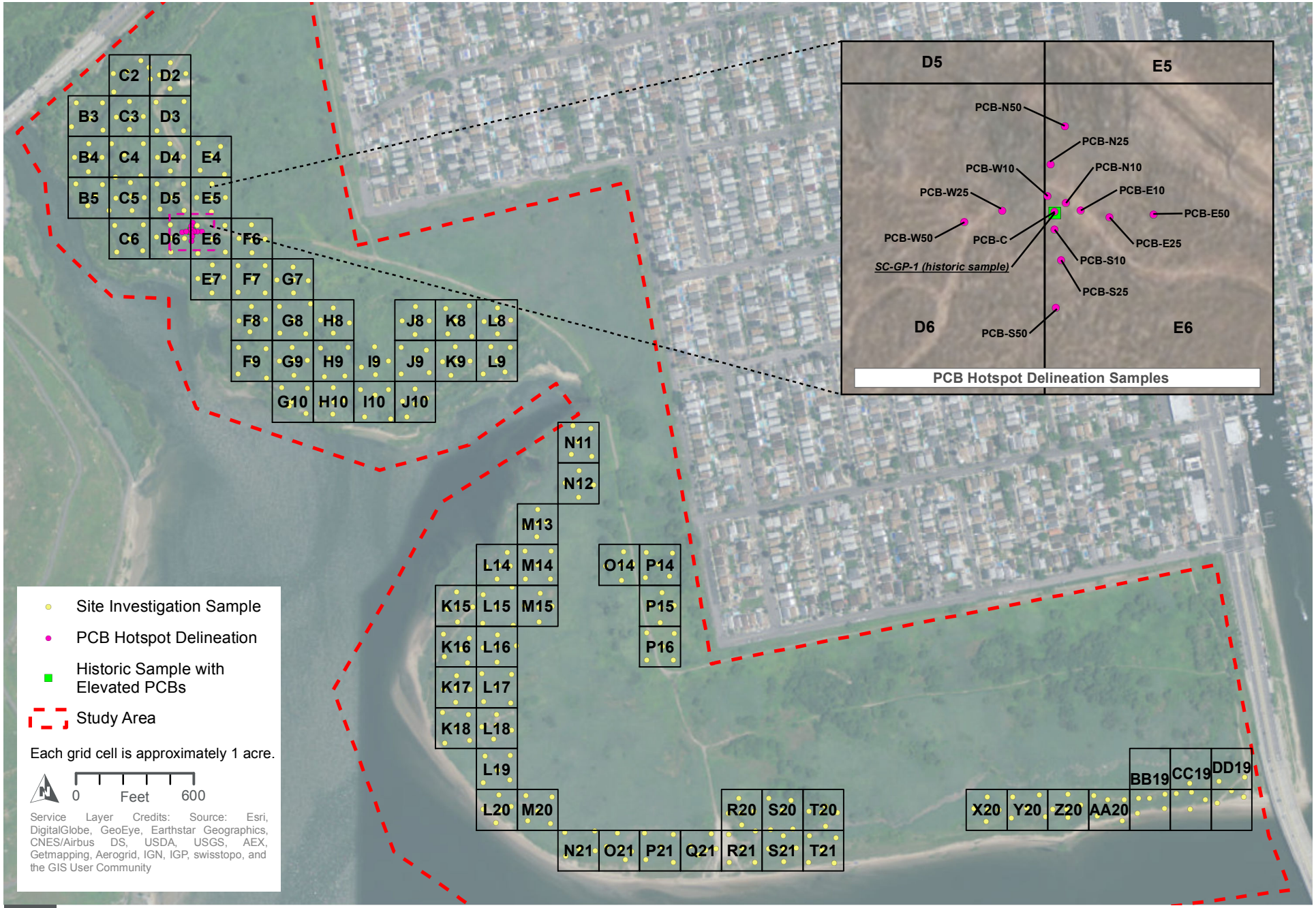
Spring Creek South | Howard Beach Site Investigation Report and Screening Level Ecological Risk Assessment (USACE, 2017a)



PROPOSED SAMPLE LOCATIONS

SPRING CREEK SOUTH

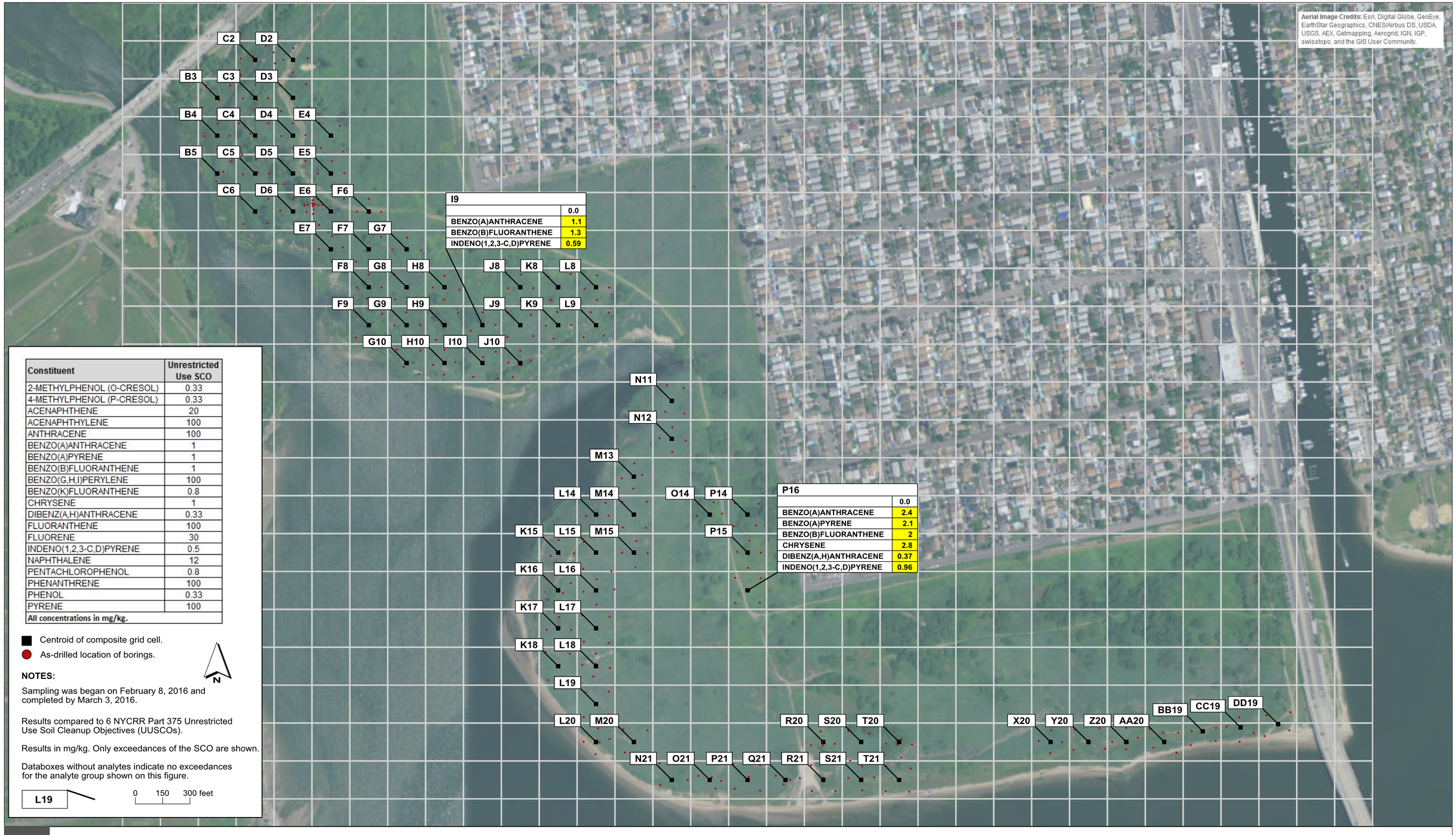
FIGURE 3-1



AS-DRILLED SAMPLE LOCATIONS

SPRING CREEK SOUTH

FIGURE 3-2



Constituent	Unrestricted Use SCO
2-METHYLPHENOL (O-CRESOL)	0.33
4-METHYLPHENOL (P-CRESOL)	0.33
ACENAPHTHENE	20
ACENAPHTHYLENE	100
ANTHRACENE	100
BENZO(A)ANTHRACENE	1
BENZO(A)PYRENE	1
BENZO(B)FLUORANTHENE	1
BENZO(G,H,I)PERYLENE	100
BENZO(K)FLUORANTHENE	0.8
CHRYSENE	1
DIBENZ(A,H)ANTHRACENE	0.33
FLUORANTHENE	100
FLUORENE	30
INDENO(1,2,3-C,D)PYRENE	0.5
NAPHTHALENE	12
PENTACHLOROPHENOL	0.8
PHENANTHRENE	100
PHENOL	0.33
PYRENE	100
All concentrations in mg/kg.	

■ Centroid of composite grid cell.

● As-drilled location of borings.

N

NOTES:

Sampling was began on February 8, 2016 and completed by March 3, 2016.

Results compared to 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives (UUSCOs).

Results in mg/kg. Only exceedances of the SCO are shown.

Databoxes without analytes indicate no exceedances for the analyte group shown on this figure.

L19

0 150 300 feet

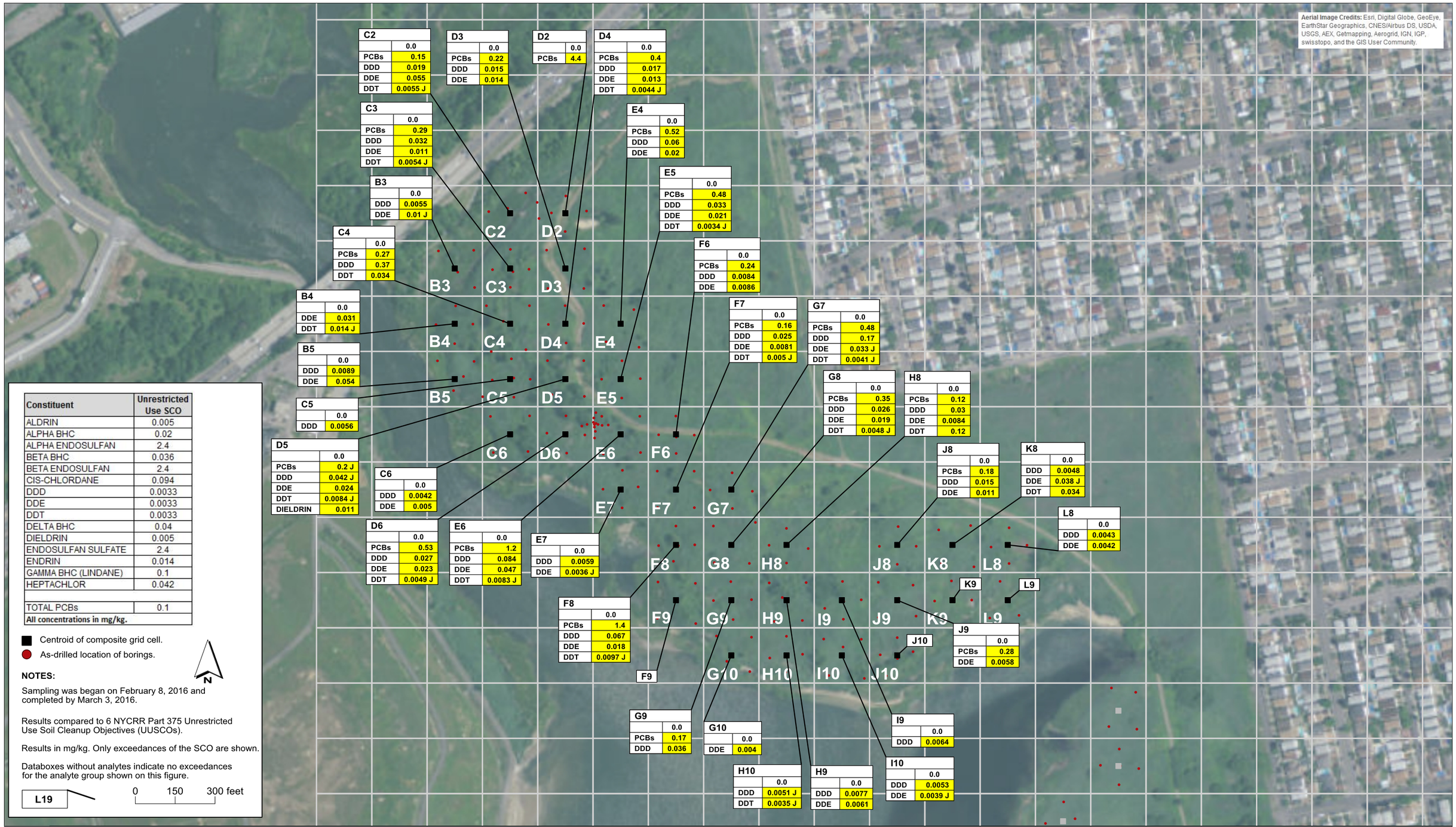
HDR

SVOC EXCEEDANCES OF UUSCOs - CUT LAYER

SPRING CREEK SOUTH

FIGURE 4-1

SPRING CREEK SOUTH SITE INVESTIGATION REPORT



Constituent	Unrestricted Use SCO
ALDRIN	0.005
ALPHA BHC	0.02
ALPHA ENDOSULFAN	2.4
BETA BHC	0.036
BETA ENDOSULFAN	2.4
CIS-CHLORDANE	0.094
DDD	0.0033
DDE	0.0033
DDT	0.0033
DELTA BHC	0.04
DIELDRIN	0.005
ENDOSULFAN SULFATE	2.4
ENDRIN	0.014
GAMMA BHC (LINDANE)	0.1
HEPTACHLOR	0.042
TOTAL PCBs	0.1

All concentrations in mg/kg.

■ Centroid of composite grid cell.

● As-drilled location of borings.

N

NOTES:

Sampling was began on February 8, 2016 and completed by March 3, 2016.

Results compared to 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives (UUSCOs).

Results in mg/kg. Only exceedances of the SCO are shown.

Databoxes without analytes indicate no exceedances for the analyte group shown on this figure.

L19

0 150 300 feet

HDR

PESTICIDE / PCB EXCEEDANCES OF UUSCOs - CUT LAYER NORTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-2

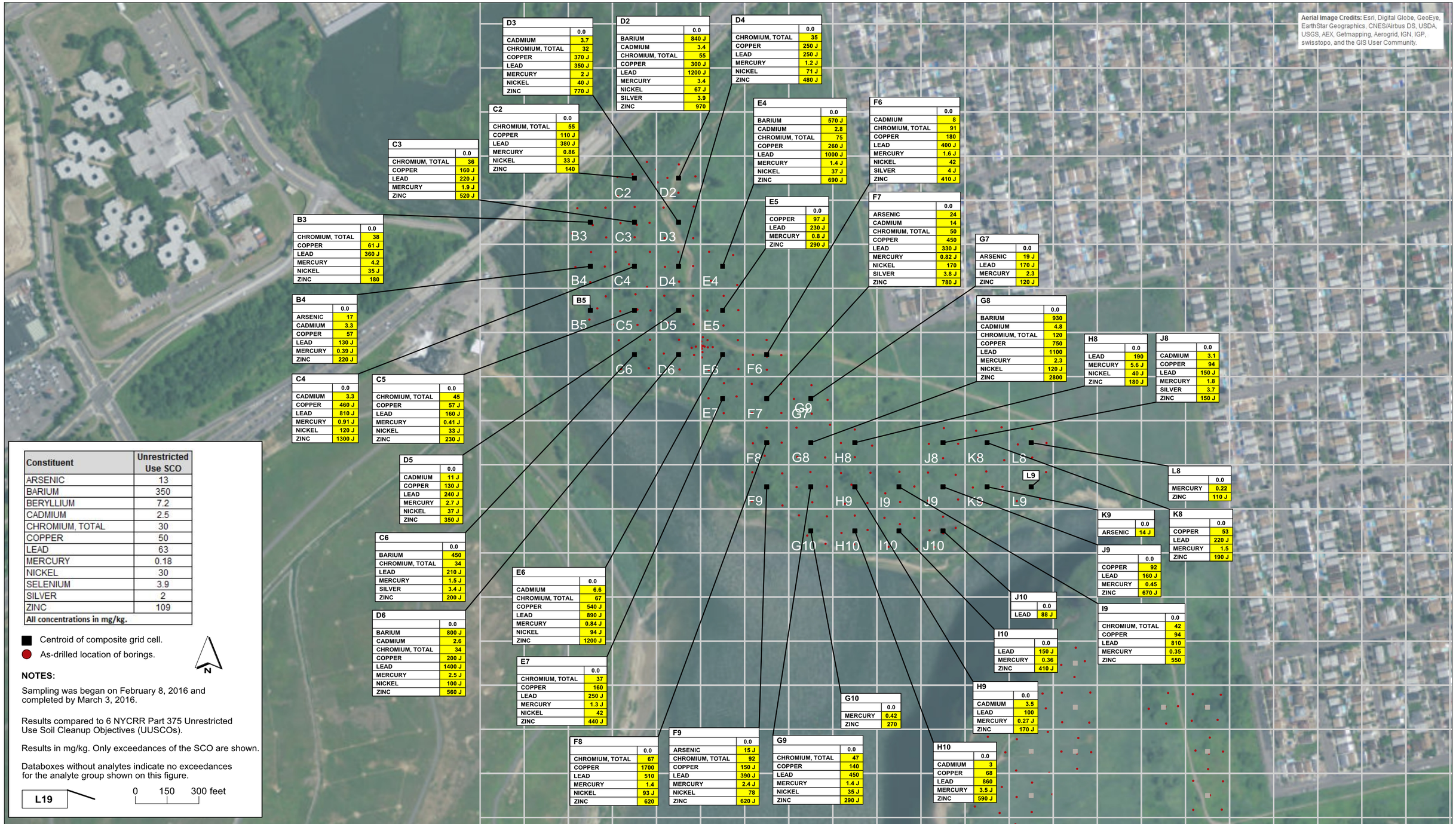
SPRING CREEK SOUTH SITE INVESTIGATION REPORT

PESTICIDE / PCB EXCEEDANCES OF UUSCOs - CUT LAYER SOUTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-3

SPRING CREEK SOUTH SITE INVESTIGATION REPORT



D3	
	0.0
CADMIUM	3.7
CHROMIUM, TOTAL	32
COPPER	370 J
LEAD	350 J
MERCURY	2 J
NICKEL	40 J
ZINC	770 J

D2	
	0.0
BARIUM	840 J
CADMIUM	3.4
CHROMIUM, TOTAL	55
COPPER	300 J
LEAD	1200 J
MERCURY	3.4
NICKEL	67 J
SILVER	3.9
ZINC	970

D4	
	0.0
CHROMIUM, TOTAL	35
COPPER	250 J
LEAD	250 J
MERCURY	1.2 J
NICKEL	71 J
ZINC	480 J

C2	
	0.0
CHROMIUM, TOTAL	55
COPPER	110 J
LEAD	380 J
MERCURY	0.86
NICKEL	33 J
ZINC	140

C3	
	0.0
CHROMIUM, TOTAL	36
COPPER	160 J
LEAD	220 J
MERCURY	1.9 J
ZINC	520 J

E4	
	0.0
BARIUM	570 J
CADMIUM	2.8
CHROMIUM, TOTAL	75
COPPER	260 J
LEAD	1000 J
MERCURY	1.4 J
NICKEL	37 J
ZINC	690 J

F6	
	0.0
CADMIUM	8
CHROMIUM, TOTAL	91
COPPER	180
LEAD	400 J
MERCURY	1.6 J
NICKEL	42
SILVER	4 J
ZINC	410 J

B3	
	0.0
CHROMIUM, TOTAL	38
COPPER	61 J
LEAD	360 J
MERCURY	4.2
NICKEL	35 J
ZINC	180

E5	
	0.0
COPPER	97 J
LEAD	230 J
MERCURY	0.8 J
ZINC	290 J

F7	
	0.0
ARSENIC	24
CADMIUM	14
CHROMIUM, TOTAL	50
COPPER	450
LEAD	330 J
MERCURY	0.82 J
NICKEL	170
SILVER	3.8 J
ZINC	780 J

G7	
	0.0
ARSENIC	19 J
LEAD	170 J
MERCURY	2.3
ZINC	120 J

B4	
	0.0
ARSENIC	17
CADMIUM	3.3
COPPER	57
LEAD	130 J
MERCURY	0.39 J
ZINC	220 J

G8	
	0.0
BARIUM	930
CADMIUM	4.8
CHROMIUM, TOTAL	120
COPPER	750
LEAD	1100
MERCURY	2.3
NICKEL	120 J
ZINC	2800

H8	
	0.0
LEAD	190
MERCURY	5.6 J
NICKEL	40 J
ZINC	180 J

J8	
	0.0
CADMIUM	3.1
COPPER	94
LEAD	150 J
MERCURY	1.8
SILVER	3.7
ZINC	150 J

C4	
	0.0
CADMIUM	3.3
COPPER	460 J
LEAD	810 J
MERCURY	0.91 J
NICKEL	120 J
ZINC	1300 J

C5	
	0.0
CHROMIUM, TOTAL	45
COPPER	57 J
LEAD	160 J
MERCURY	0.41 J
NICKEL	33 J
ZINC	230 J

D5	
	0.0
CADMIUM	11 J
COPPER	130 J
LEAD	240 J
MERCURY	2.7 J
NICKEL	37 J
ZINC	350 J

C6	
	0.0
BARIUM	450
CHROMIUM, TOTAL	34
LEAD	210 J
MERCURY	1.5 J
SILVER	3.4 J
ZINC	200 J

D6	
	0.0
BARIUM	800 J
CADMIUM	2.6
CHROMIUM, TOTAL	34
COPPER	200 J
LEAD	1400 J
MERCURY	2.5 J
NICKEL	100 J
ZINC	560 J

E6	
	0.0
CADMIUM	6.6
CHROMIUM, TOTAL	67
COPPER	540 J
LEAD	890 J
MERCURY	0.84 J
NICKEL	94 J
ZINC	1200 J

E7	
	0.0
CHROMIUM, TOTAL	37
COPPER	160
LEAD	250 J
MERCURY	1.3 J
NICKEL	42
ZINC	440 J

F8	
	0.0
CHROMIUM, TOTAL	67
COPPER	1700
LEAD	510
MERCURY	1.4
NICKEL	93 J
ZINC	620

F9	
	0.0
ARSENIC	15 J
CHROMIUM, TOTAL	92
COPPER	150 J
LEAD	390 J
MERCURY	2.4 J
NICKEL	78
ZINC	620 J

G9	
	0.0
CHROMIUM, TOTAL	47
COPPER	140
LEAD	450
MERCURY	1.4 J
NICKEL	35 J
ZINC	290 J

G10	
	0.0
MERCURY	0.42
ZINC	270

H10	
	0.0
CADMIUM	3
COPPER	68
LEAD	860
MERCURY	3.5 J
ZINC	590 J

H9	
	0.0
CADMIUM	3.5
LEAD	100
MERCURY	0.27 J
ZINC	170 J

I10	
	0.0
LEAD	150 J
MERCURY	0.36
ZINC	410 J

I9	
	0.0
CHROMIUM, TOTAL	42
COPPER	94
LEAD	810
MERCURY	0.35
ZINC	550

J9	
	0.0
COPPER	92
LEAD	160 J
MERCURY	0.45
ZINC	670 J

J10	
	0.0
LEAD	88 J

K9	
	0.0
ARSENIC	14 J

K8	
	0.0
COPPER	53
LEAD	220 J
MERCURY	1.5
ZINC	190 J

L8	
	0.0
MERCURY	0.22
ZINC	110 J

Constituent	Unrestricted Use SCO
ARSENIC	13
BARIUM	350
BERYLLIUM	7.2
CADMIUM	2.5
CHROMIUM, TOTAL	30
COPPER	50
LEAD	63
MERCURY	0.18
NICKEL	30
SELENIUM	3.9
SILVER	2
ZINC	109
All concentrations in mg/kg.	

- Centroid of composite grid cell.
- As-drilled location of borings.

NOTES:

Sampling was began on February 8, 2016 and completed by March 3, 2016.

Results compared to 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives (UUSCOs).

Results in mg/kg. Only exceedances of the SCO are shown.

Databoxes without analytes indicate no exceedances for the analyte group shown on this figure.

INORGANIC EXCEEDANCES OF UUSCOs - CUT LAYER NORTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-4

SPRING CREEK SOUTH SITE INVESTIGATION REPORT

Constituent	Unrestricted Use SCO
ARSENIC	13
BARIUM	350
BERYLLIUM	7.2
CADMIUM	2.5
CHROMIUM, TOTAL	30
COPPER	50
LEAD	63
MERCURY	0.18
NICKEL	30
SELENIUM	3.9
SILVER	2
ZINC	109
All concentrations in mg/kg.	

N11	0.0
COPPER	120
LEAD	200 J
MERCURY	2.2
NICKEL	31 J
SILVER	5.1
ZINC	210 J

N12	0.0
BARIUM	460
CADMIUM	16
CHROMIUM, TOTAL	310
COPPER	660
LEAD	670 J
MERCURY	40
NICKEL	51 J
SILVER	25
ZINC	550 J

M13	0.0
CADMIUM	4
CHROMIUM, TOTAL	46 J
COPPER	350
LEAD	9700
MERCURY	1.9 J
NICKEL	71 J
SILVER	6.2 J
ZINC	960

M14	0.0
BARIUM	380 J
CADMIUM	6.1
CHROMIUM, TOTAL	39 J
COPPER	210
LEAD	740
MERCURY	2.2 J
NICKEL	42 J
SILVER	2.1 J
ZINC	810

O14	0.0
BARIUM	740 J
CADMIUM	5.3
CHROMIUM, TOTAL	54 J
COPPER	200 J
LEAD	310 J
MERCURY	4.9 J
SILVER	7.1 J
ZINC	590

P14	0.0
COPPER	83
LEAD	230
MERCURY	1.1 J
ZINC	150

M15	0.0
BARIUM	510 J
CHROMIUM, TOTAL	76 J
COPPER	260
LEAD	440
MERCURY	5.7 J
NICKEL	62 J
SILVER	5.6 J
ZINC	1900

L15	0.0
BARIUM	530 J
COPPER	70
LEAD	870
MERCURY	1.1 J
ZINC	380

L14	0.0
MERCURY	0.74 J

K16	0.0
SILVER	2.8 J
ZINC	110

L16	0.0
BARIUM	430 J
CHROMIUM, TOTAL	41 J
COPPER	370
LEAD	600
MERCURY	8.6 J
NICKEL	45 J
ZINC	4000

K15	0.0

P15	0.0
CHROMIUM, TOTAL	31 J
COPPER	72
LEAD	270
MERCURY	0.83 J
ZINC	220

K17	0.0
COPPER	63
LEAD	180
MERCURY	1.5
ZINC	360

L18	0.0
BARIUM	2400
CADMIUM	4.5
CHROMIUM, TOTAL	1000 J
COPPER	170
LEAD	300
MERCURY	0.38
NICKEL	59
ZINC	1300

P16	0.0
COPPER	93
LEAD	270
MERCURY	0.65 J
ZINC	200

L17	0.0
ARSENIC	44
BARIUM	460
CADMIUM	3
CHROMIUM, TOTAL	37 J
COPPER	560
LEAD	320
MERCURY	1
NICKEL	41
ZINC	640

R20	0.0
COPPER	85 J
LEAD	83
MERCURY	1.6
SILVER	2.8

Y20	0.0
COPPER	66 J
LEAD	150
MERCURY	0.64

AA20	0.0
MERCURY	0.37

K18	0.0
COPPER	77
LEAD	200
MERCURY	1
ZINC	380

L19	0.0
BARIUM	380
COPPER	120
LEAD	450
MERCURY	3.6
ZINC	270

T20	0.0
CHROMIUM, TOTAL	53 J
COPPER	130 J
LEAD	300
MERCURY	3.9
ZINC	270 J

L20	0.0
COPPER	150
LEAD	670
MERCURY	1.2
ZINC	460

M20	0.0
CHROMIUM, TOTAL	38 J
COPPER	140 J
LEAD	690
MERCURY	2.4
NICKEL	110
ZINC	510 J

N21	0.0
LEAD	240
MERCURY	0.88
ZINC	200 J

O21	0.0
CHROMIUM, TOTAL	41 J
COPPER	160 J
LEAD	8000
MERCURY	2.8
NICKEL	31
ZINC	470 J

P21	0.0
CHROMIUM, TOTAL	36 J
COPPER	110 J
LEAD	350
MERCURY	1.1
ZINC	340 J

S21	0.0
MERCURY	0.25

S20	0.0
CADMIUM	3.3 J
CHROMIUM, TOTAL	61 J
COPPER	340 J
LEAD	500
MERCURY	4.6
NICKEL	46
SILVER	4.2
ZINC	610 J

X20	0.0
COPPER	83 J
LEAD	150
MERCURY	1
ZINC	270

Z20	0.0
MERCURY	0.47

BB19	0.0
MERCURY	0.2

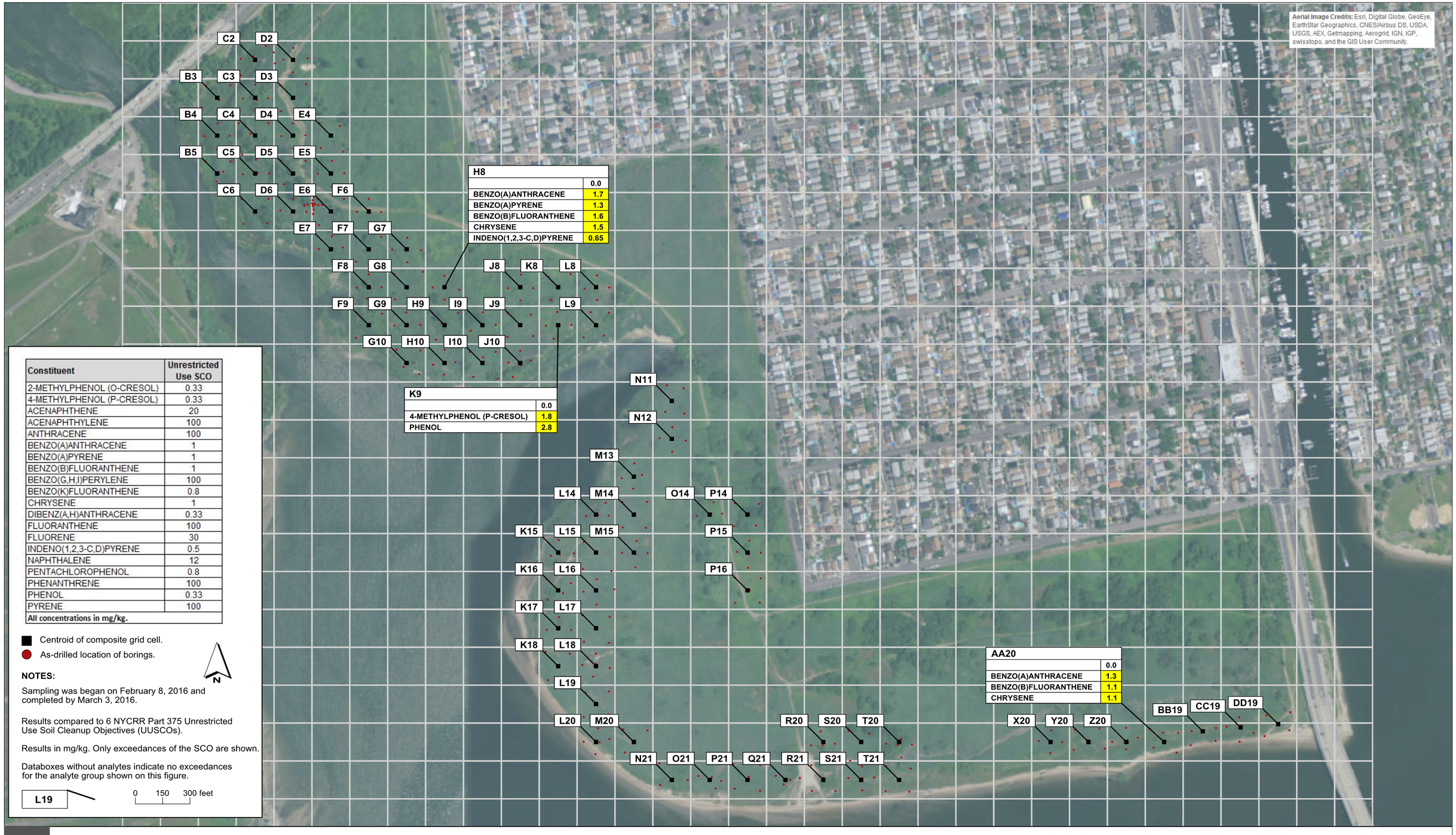
HDR

INORGANIC EXCEEDANCES OF UUSCOs - CUT LAYER SOUTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-5

SPRING CREEK SOUTH SITE INVESTIGATION REPORT



Constituent	Unrestricted Use SCO
2-METHYLPHENOL (O-CRESOL)	0.33
4-METHYLPHENOL (P-CRESOL)	0.33
ACENAPHTHENE	20
ACENAPHTHYLENE	100
ANTHRACENE	100
BENZO(A)ANTHRACENE	1
BENZO(A)PYRENE	1
BENZO(B)FLUORANTHENE	1
BENZO(G,H,I)PERYLENE	100
BENZO(K)FLUORANTHENE	0.8
CHRYSENE	1
DIBENZ(A,H)ANTHRACENE	0.33
FLUORANTHENE	100
FLUORENE	30
INDENO(1,2,3-C,D)PYRENE	0.5
NAPHTHALENE	12
PENTACHLOROPHENOL	0.8
PHENANTHRENE	100
PHENOL	0.33
PYRENE	100
All concentrations in mg/kg.	

■ Centroid of composite grid cell.

● As-drilled location of borings.

N

NOTES:

Sampling was began on February 8, 2016 and completed by March 3, 2016.

Results compared to 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives (UUSCOs).

Results in mg/kg. Only exceedances of the SCO are shown.

Databoxes without analytes indicate no exceedances for the analyte group shown on this figure.

L19

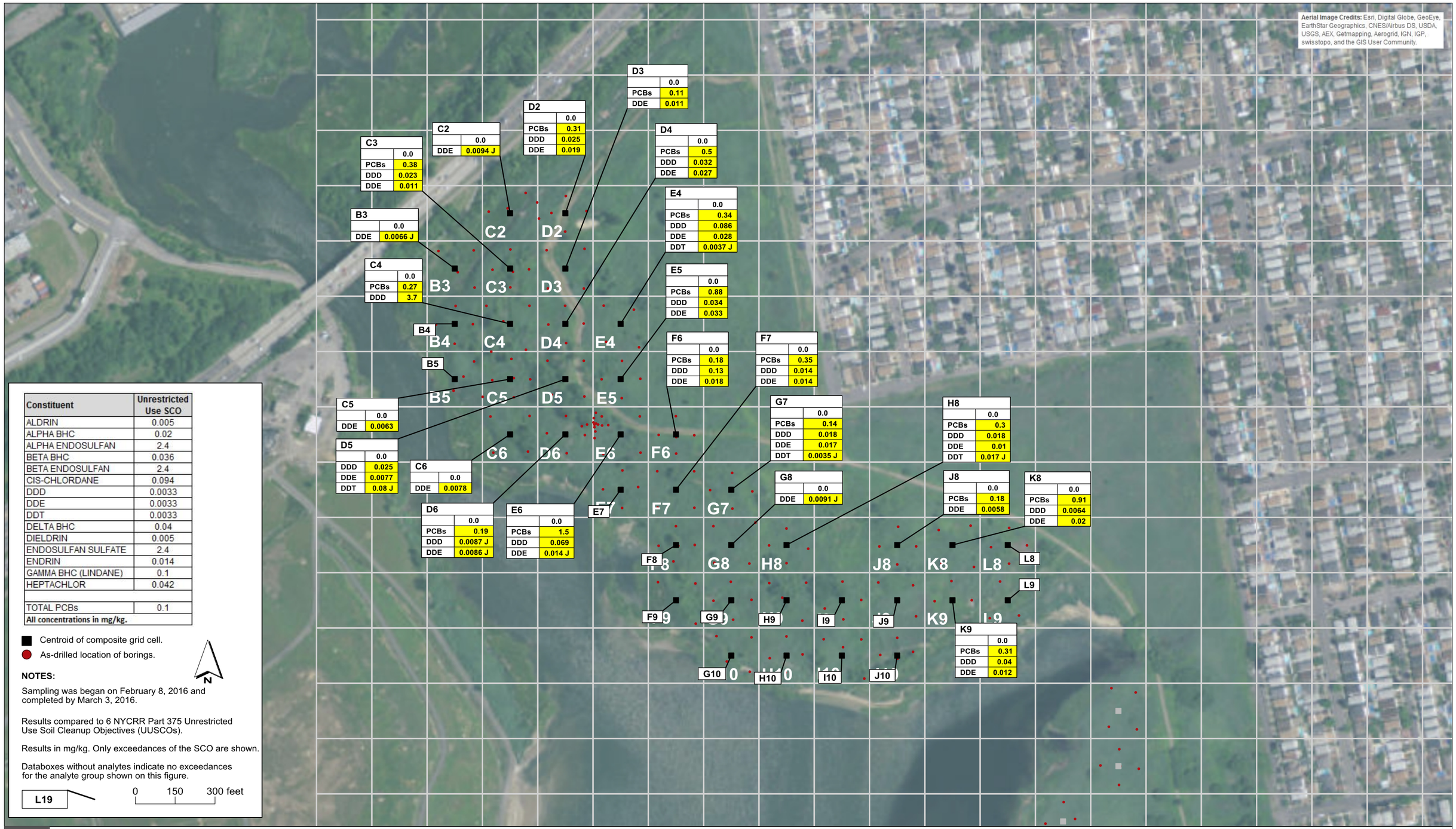
0 150 300 feet

SVOC EXCEEDANCES OF USSCOs - EXPOSED LAYER

SPRING CREEK SOUTH

FIGURE 4-6

SPRING CREEK SOUTH SITE INVESTIGATION REPORT



Constituent	Unrestricted Use SCO
ALDRIN	0.005
ALPHA BHC	0.02
ALPHA ENDOSULFAN	2.4
BETA BHC	0.036
BETA ENDOSULFAN	2.4
CIS-CHLORDANE	0.094
DDD	0.0033
DDE	0.0033
DDT	0.0033
DELTA BHC	0.04
DIELDRIN	0.005
ENDOSULFAN SULFATE	2.4
ENDRIN	0.014
GAMMA BHC (LINDANE)	0.1
HEPTACHLOR	0.042
TOTAL PCBs	0.1

All concentrations in mg/kg.

■ Centroid of composite grid cell.

● As-drilled location of borings.

N

NOTES:

Sampling was began on February 8, 2016 and completed by March 3, 2016.

Results compared to 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives (UUSCOs).

Results in mg/kg. Only exceedances of the SCO are shown.

Databoxes without analytes indicate no exceedances for the analyte group shown on this figure.

L19

0 150 300 feet

HDR

PESTICIDE / PCB EXCEEDANCES OF UUSCOs - EXPOSED LAYER NORTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-7

SPRING CREEK SOUTH SITE INVESTIGATION REPORT

Constituent	Unrestricted Use SCO
ALDRIN	0.005
ALPHA BHC	0.02
ALPHA ENDOSULFAN	2.4
BETA BHC	0.036
BETA ENDOSULFAN	2.4
CIS-CHLORDANE	0.094
DDD	0.0033
DDE	0.0033
DDT	0.0033
DELTA BHC	0.04
DIELDRIN	0.005
ENDOSULFAN SULFATE	2.4
ENDRIN	0.014
GAMMA BHC (LINDANE)	0.1
HEPTACHLOR	0.042
TOTAL PCBs	0.1

All concentrations in mg/kg.

N11	0.0

N12	0.0
DDD	0.032
DDE	0.0037 J
DDT	0.0046 J

M13	0.0
DDD	0.0065

M14	0.0
DDD	0.0037
DDE	0.0046 J
DDT	0.0047

O14	0.0
PCBs	0.55
DDD	17

P14	0.0

L14	0.0

L16	0.0
PCBs	0.12
DDD	0.28
DDE	0.11
DDT	0.1

K15	0.0

L15	0.0

M15	0.0
PCBs	0.51
DDD	0.12 J
DDE	0.017 J
DDT	0.014 J

P15	0.0

K16	0.0

P16	0.0
DDD	0.0048
DDE	0.0034 J

K17	0.0

L17	0.0
DDD	0.0063
DDE	0.0036 J

K18	0.0

L18	0.0
DDD	0.077
DDE	0.027 J
DDT	0.49

L19	0.0

L20	0.0

M20	0.0
DDD	0.059
DDE	0.0057

Q21	0.0
DDD	0.0044
DDE	0.0038 J

R20	0.0
DDD	0.01
DDE	0.0045 J

S20	0.0
PCBs	0.41
DDD	0.076
DDE	0.009 J
DDT	0.011 J

T20	0.0
PCBs	0.12
DDD	0.068
DDE	0.0068
DDT	0.014 J

X20	0.0
DDD	0.28

Y20	0.0
DDD	0.028

N21	0.0

O21	0.0

P21	0.0
DDD	0.045 J
DDE	0.0058 J
DDT	0.0045 J

R21	0.0

S21	0.0
PCBs	0.18
DDD	0.017

Z20	0.0

AA20	0.0

BB19	0.0

CC19	0.0

DD19	0.0

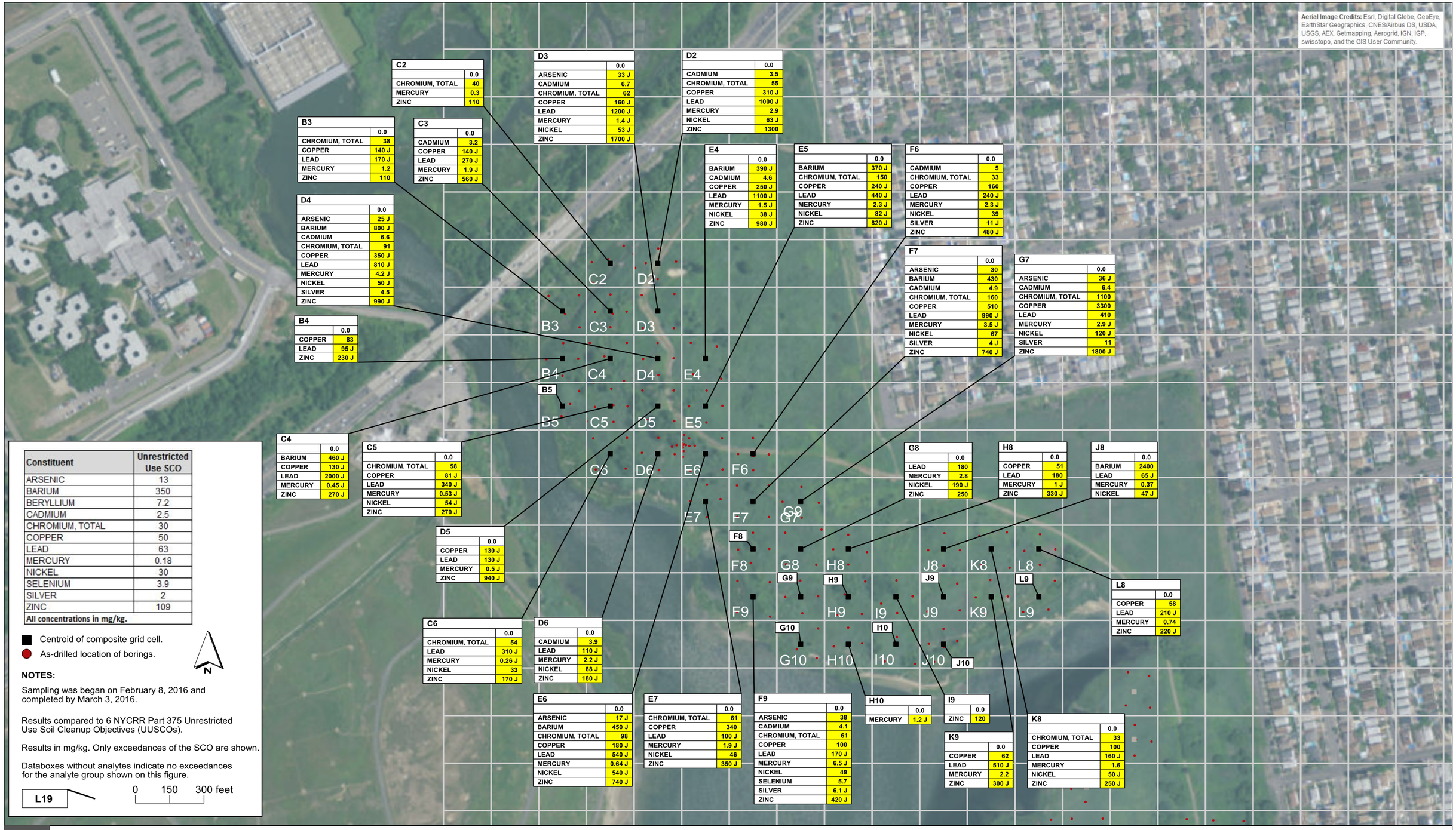
HDR

PESTICIDE / PCB EXCEEDANCES OF UUSCOs - EXPOSED LAYER SOUTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-8

SPRING CREEK SOUTH SITE INVESTIGATION REPORT



Constituent	Unrestricted Use SCO
ARSENIC	13
BARIUM	350
BERYLLIUM	7.2
CADMIUM	2.5
CHROMIUM, TOTAL	30
COPPER	50
LEAD	63
MERCURY	0.18
NICKEL	30
SELENIUM	3.9
SILVER	2
ZINC	109
All concentrations in mg/kg.	

- Centroid of composite grid cell.
- As-drilled location of borings.

N

NOTES:

Sampling was began on February 8, 2016 and completed by March 3, 2016.

Results compared to 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives (UUSCOs).

Results in mg/kg. Only exceedances of the SCO are shown.

Databoxes without analytes indicate no exceedances for the analyte group shown on this figure.

L19

0 150 300 feet

C2	0.0
CHROMIUM, TOTAL	40
MERCURY	0.3
ZINC	110

D3	0.0
ARSENIC	33 J
CADMIUM	6.7
CHROMIUM, TOTAL	62
COPPER	160 J
LEAD	1200 J
MERCURY	1.4 J
NICKEL	53 J
ZINC	1700 J

D2	0.0
CADMIUM	3.5
CHROMIUM, TOTAL	55
COPPER	310 J
LEAD	1000 J
MERCURY	2.9
NICKEL	63 J
ZINC	1300

B3	0.0
CHROMIUM, TOTAL	38
COPPER	140 J
LEAD	170 J
MERCURY	1.2
ZINC	110

C3	0.0
CADMIUM	3.2
COPPER	140 J
LEAD	270 J
MERCURY	1.9 J
ZINC	560 J

E4	0.0
BARIUM	390 J
CADMIUM	4.6
COPPER	250 J
LEAD	1100 J
MERCURY	1.5 J
NICKEL	38 J
ZINC	980 J

E5	0.0
BARIUM	370 J
CHROMIUM, TOTAL	150
COPPER	240 J
LEAD	440 J
MERCURY	2.3 J
NICKEL	82 J
ZINC	820 J

F6	0.0
CADMIUM	5
CHROMIUM, TOTAL	33
COPPER	160
LEAD	240 J
MERCURY	2.3 J
NICKEL	39
SILVER	11 J
ZINC	480 J

D4	0.0
ARSENIC	25 J
BARIUM	800 J
CADMIUM	6.6
CHROMIUM, TOTAL	91
COPPER	350 J
LEAD	810 J
MERCURY	4.2 J
NICKEL	50 J
SILVER	4.5
ZINC	990 J

F7	0.0
ARSENIC	30
BARIUM	430
CADMIUM	4.9
CHROMIUM, TOTAL	160
COPPER	510
LEAD	990 J
MERCURY	3.5 J
NICKEL	67
SILVER	4 J
ZINC	740 J

G7	0.0
ARSENIC	36 J
CADMIUM	6.4
CHROMIUM, TOTAL	1100
COPPER	3300
LEAD	410
MERCURY	2.9 J
NICKEL	120 J
SILVER	11
ZINC	1800 J

B4	0.0
COPPER	83
LEAD	95 J
ZINC	230 J

C4	0.0
BARIUM	460 J
COPPER	130 J
LEAD	2000 J
MERCURY	0.45 J
ZINC	270 J

C5	0.0
CHROMIUM, TOTAL	58
COPPER	81 J
LEAD	340 J
MERCURY	0.53 J
NICKEL	54 J
ZINC	270 J

G8	0.0
LEAD	180
MERCURY	2.8
NICKEL	190 J
ZINC	250

H8	0.0
COPPER	51
LEAD	180
MERCURY	1 J
ZINC	330 J

J8	0.0
BARIUM	2400
LEAD	65 J
MERCURY	0.37
NICKEL	47 J

D5	0.0
COPPER	130 J
LEAD	130 J
MERCURY	0.5 J
ZINC	940 J

L8	0.0
COPPER	58
LEAD	210 J
MERCURY	0.74
ZINC	220 J

C6	0.0
CHROMIUM, TOTAL	54
LEAD	310 J
MERCURY	0.26 J
NICKEL	33
ZINC	170 J

D6	0.0
CADMIUM	3.9
LEAD	110 J
MERCURY	2.2 J
NICKEL	88 J
ZINC	180 J

E6	0.0
ARSENIC	17 J
BARIUM	450 J
CHROMIUM, TOTAL	98
COPPER	180 J
LEAD	540 J
MERCURY	0.64 J
NICKEL	540 J
ZINC	740 J

E7	0.0
CHROMIUM, TOTAL	61
COPPER	340
LEAD	100 J
MERCURY	1.9 J
NICKEL	46
ZINC	350 J

F9	0.0
ARSENIC	38
CADMIUM	4.1
CHROMIUM, TOTAL	61
COPPER	100
LEAD	170 J
MERCURY	6.5 J
NICKEL	49
SELENIUM	5.7
SILVER	6.1 J
ZINC	420 J

H10	0.0
MERCURY	1.2 J

I9	0.0
ZINC	120

K9	0.0
COPPER	62
LEAD	510 J
MERCURY	2.2
ZINC	300 J

K8	0.0
CHROMIUM, TOTAL	33
COPPER	100
LEAD	160 J
MERCURY	1.6
NICKEL	50 J
ZINC	250 J

INORGANIC EXCEEDANCES OF UUSCOs - EXPOSED LAYER NORTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-9

SPRING CREEK SOUTH SITE INVESTIGATION REPORT

N11	0.0
COPPER	120
LEAD	200 J
MERCURY	2.2
NICKEL	31 J
SILVER	5.1
ZINC	210 J

N12	0.0
BARIUM	460
CADMIUM	16
CHROMIUM, TOTAL	310
COPPER	660
LEAD	670 J
MERCURY	40
NICKEL	51 J
SILVER	25
ZINC	550 J

M13	0.0
CADMIUM	4
CHROMIUM, TOTAL	46 J
COPPER	350
LEAD	9700
MERCURY	1.9 J
NICKEL	71 J
SILVER	6.2 J
ZINC	960

M14	0.0
BARIUM	380 J
CADMIUM	6.1
CHROMIUM, TOTAL	39 J
COPPER	210
LEAD	740
MERCURY	2.2 J
NICKEL	42 J
SILVER	2.1 J
ZINC	810

O14	0.0
BARIUM	740 J
CADMIUM	5.3
CHROMIUM, TOTAL	54 J
COPPER	200 J
LEAD	310 J
MERCURY	4.9 J
SILVER	7.1 J
ZINC	590

P14	0.0
COPPER	83
LEAD	230
MERCURY	1.1 J
ZINC	150

M15	0.0
BARIUM	510 J
CHROMIUM, TOTAL	76 J
COPPER	260
LEAD	440
MERCURY	5.7 J
NICKEL	62 J
SILVER	5.6 J
ZINC	1900

L15	0.0
BARIUM	530 J
COPPER	70
LEAD	870
MERCURY	1.1 J
ZINC	380

L14	0.0
MERCURY	0.74 J

K16	0.0
SILVER	2.8 J
ZINC	110

L16	0.0
BARIUM	430 J
CHROMIUM, TOTAL	41 J
COPPER	370
LEAD	600
MERCURY	8.6 J
NICKEL	45 J
ZINC	4000

K15

P15	0.0
CHROMIUM, TOTAL	31 J
COPPER	72
LEAD	270
MERCURY	0.83 J
ZINC	220

L18	0.0
BARIUM	2400
CADMIUM	4.5
CHROMIUM, TOTAL	1000 J
COPPER	170
LEAD	300
MERCURY	0.38
NICKEL	59
ZINC	1300

P16	0.0
COPPER	93
LEAD	270
MERCURY	0.65 J
ZINC	200

K17	0.0
COPPER	63
LEAD	180
MERCURY	1.5
ZINC	360

L17	0.0
ARSENIC	44
BARIUM	460
CADMIUM	3
CHROMIUM, TOTAL	37 J
COPPER	560
LEAD	320
MERCURY	1
NICKEL	41
ZINC	640

K18	0.0
COPPER	77
LEAD	200
MERCURY	1
ZINC	380

L19	0.0
BARIUM	380
COPPER	120
LEAD	450
MERCURY	3.6
ZINC	270

R20	0.0
COPPER	85 J
LEAD	83
MERCURY	1.6
SILVER	2.8

T20	0.0
CHROMIUM, TOTAL	53 J
COPPER	130 J
LEAD	300
MERCURY	3.9
ZINC	270 J

Y20	0.0
COPPER	66 J
LEAD	150
MERCURY	0.64

AA20	0.0
MERCURY	0.37

L20	0.0
COPPER	150
LEAD	670
MERCURY	1.2
ZINC	460

M20	0.0
CHROMIUM, TOTAL	38 J
COPPER	140 J
LEAD	690
MERCURY	2.4
NICKEL	110
ZINC	510 J

N21	0.0
LEAD	240
MERCURY	0.88
ZINC	200 J

O21	0.0
CHROMIUM, TOTAL	41 J
COPPER	160 J
LEAD	8000
MERCURY	2.8
NICKEL	31
ZINC	470 J

P21	0.0
CHROMIUM, TOTAL	36 J
COPPER	110 J
LEAD	350
MERCURY	1.1
ZINC	340 J

S21	0.0
MERCURY	0.25

S20	0.0
CADMIUM	3.3 J
CHROMIUM, TOTAL	61 J
COPPER	340 J
LEAD	500
MERCURY	4.6
NICKEL	46
SILVER	4.2
ZINC	610 J

X20	0.0
COPPER	83 J
LEAD	150
MERCURY	1
ZINC	270

Z20	0.0
MERCURY	0.47

BB19	0.0
MERCURY	0.2

CC19

DD19

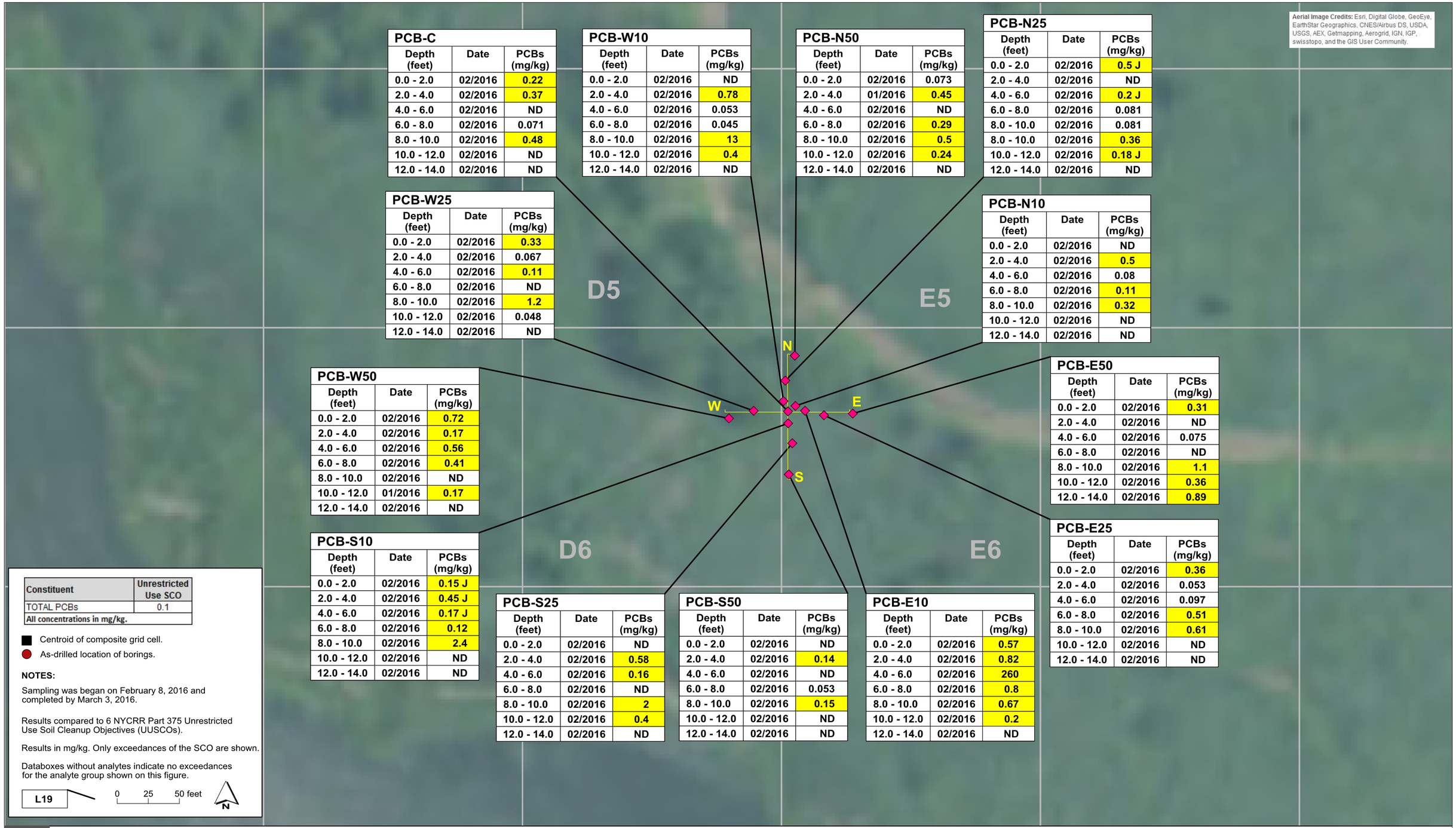
HDR

INORGANIC EXCEEDANCES OF UUSCOs - EXPOSED LAYER SOUTHERN HALF

SPRING CREEK SOUTH

FIGURE 4-10

SPRING CREEK SOUTH SITE INVESTIGATION REPORT



PCB-C

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.22
2.0 - 4.0	02/2016	0.37
4.0 - 6.0	02/2016	ND
6.0 - 8.0	02/2016	0.071
8.0 - 10.0	02/2016	0.48
10.0 - 12.0	02/2016	ND
12.0 - 14.0	02/2016	ND

PCB-W10

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	ND
2.0 - 4.0	02/2016	0.78
4.0 - 6.0	02/2016	0.053
6.0 - 8.0	02/2016	0.045
8.0 - 10.0	02/2016	13
10.0 - 12.0	02/2016	0.4
12.0 - 14.0	02/2016	ND

PCB-N50

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.073
2.0 - 4.0	01/2016	0.45
4.0 - 6.0	02/2016	ND
6.0 - 8.0	02/2016	0.29
8.0 - 10.0	02/2016	0.5
10.0 - 12.0	02/2016	0.24
12.0 - 14.0	02/2016	ND

PCB-N25

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.5 J
2.0 - 4.0	02/2016	ND
4.0 - 6.0	02/2016	0.2 J
6.0 - 8.0	02/2016	0.081
8.0 - 10.0	02/2016	0.081
8.0 - 10.0	02/2016	0.36
10.0 - 12.0	02/2016	0.18 J
12.0 - 14.0	02/2016	ND

PCB-W25

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.33
2.0 - 4.0	02/2016	0.067
4.0 - 6.0	02/2016	0.11
6.0 - 8.0	02/2016	ND
8.0 - 10.0	02/2016	1.2
10.0 - 12.0	02/2016	0.048
12.0 - 14.0	02/2016	ND

PCB-N10

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	ND
2.0 - 4.0	02/2016	0.5
4.0 - 6.0	02/2016	0.08
6.0 - 8.0	02/2016	0.11
8.0 - 10.0	02/2016	0.32
10.0 - 12.0	02/2016	ND
12.0 - 14.0	02/2016	ND

PCB-W50

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.72
2.0 - 4.0	02/2016	0.17
4.0 - 6.0	02/2016	0.56
6.0 - 8.0	02/2016	0.41
8.0 - 10.0	02/2016	ND
10.0 - 12.0	01/2016	0.17
12.0 - 14.0	02/2016	ND

PCB-E50

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.31
2.0 - 4.0	02/2016	ND
4.0 - 6.0	02/2016	0.075
6.0 - 8.0	02/2016	ND
8.0 - 10.0	02/2016	1.1
10.0 - 12.0	02/2016	0.36
12.0 - 14.0	02/2016	0.89

PCB-S10

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.15 J
2.0 - 4.0	02/2016	0.45 J
4.0 - 6.0	02/2016	0.17 J
6.0 - 8.0	02/2016	0.12
8.0 - 10.0	02/2016	2.4
10.0 - 12.0	02/2016	ND
12.0 - 14.0	02/2016	ND

PCB-E25

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.36
2.0 - 4.0	02/2016	0.053
4.0 - 6.0	02/2016	0.097
6.0 - 8.0	02/2016	0.51
8.0 - 10.0	02/2016	0.61
10.0 - 12.0	02/2016	ND
12.0 - 14.0	02/2016	ND

PCB-S25

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	ND
2.0 - 4.0	02/2016	0.58
4.0 - 6.0	02/2016	0.16
6.0 - 8.0	02/2016	ND
8.0 - 10.0	02/2016	2
10.0 - 12.0	02/2016	0.4
12.0 - 14.0	02/2016	ND

PCB-S50

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	ND
2.0 - 4.0	02/2016	0.14
4.0 - 6.0	02/2016	ND
6.0 - 8.0	02/2016	0.053
8.0 - 10.0	02/2016	0.15
10.0 - 12.0	02/2016	ND
12.0 - 14.0	02/2016	ND

PCB-E10

Depth (feet)	Date	PCBs (mg/kg)
0.0 - 2.0	02/2016	0.57
2.0 - 4.0	02/2016	0.82
4.0 - 6.0	02/2016	260
6.0 - 8.0	02/2016	0.8
8.0 - 10.0	02/2016	0.67
10.0 - 12.0	02/2016	0.2
12.0 - 14.0	02/2016	ND

Constituent	Unrestricted Use SCO
TOTAL PCBs	0.1
All concentrations in mg/kg.	

■ Centroid of composite grid cell.

● As-drilled location of borings.

NOTES:

Sampling was began on February 8, 2016 and completed by March 3, 2016.

Results compared to 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives (UUSCOs).

Results in mg/kg. Only exceedances of the SCO are shown.

Databoxes without analytes indicate no exceedances for the analyte group shown on this figure.

L19

0 25 50 feet

N

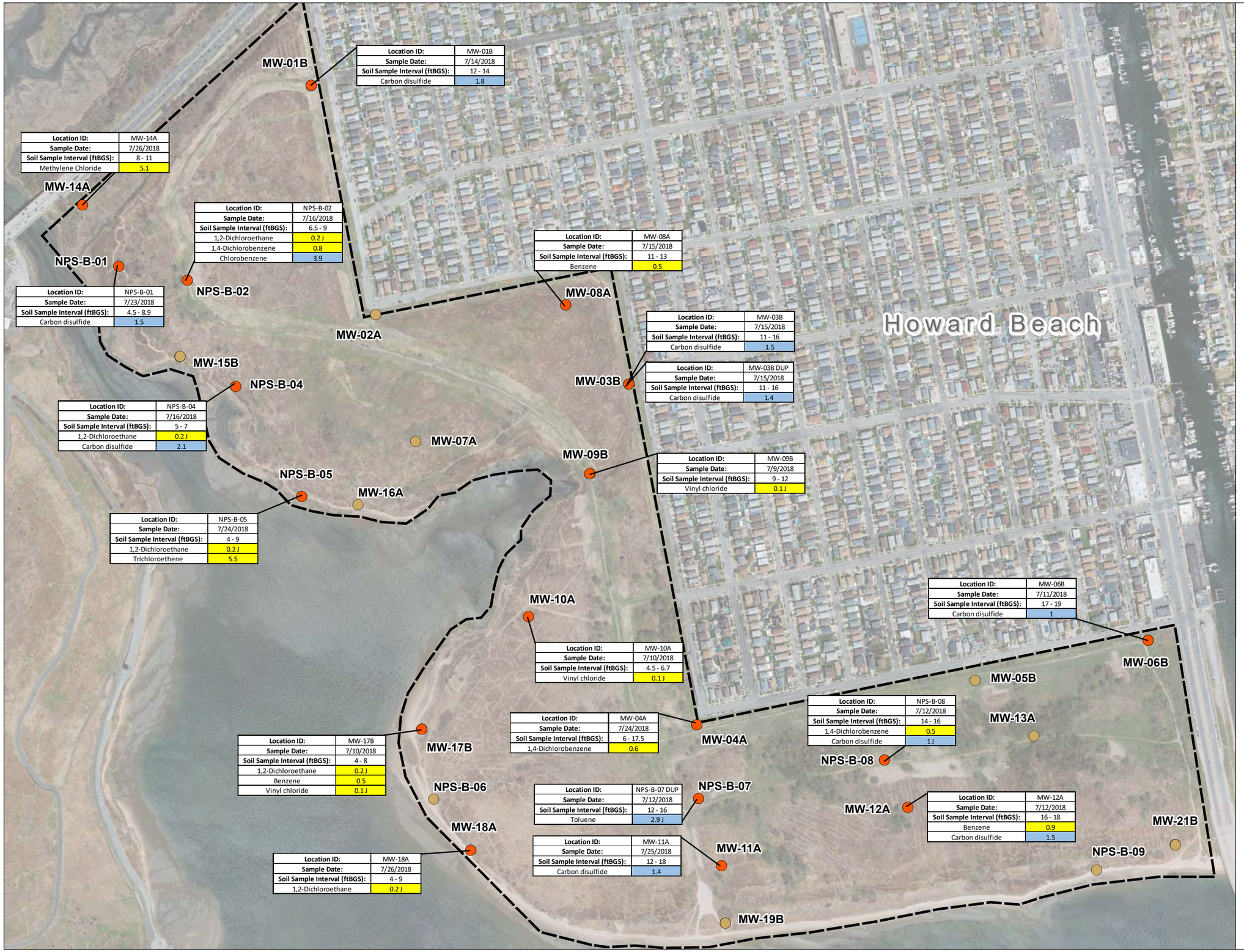
HDR

PCB DELINEATION EXCEEDANCES OF UUSCOs

SPRING CREEK SOUTH

FIGURE 4-11

Final Field Investigation Summary Report, Gateway National Recreation Area Spring Creek Park Site (JCO, 2020)



Legend

- Soil/Fill Sample - At Least One Screening Level Exceedance
- Soil/Fill Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

Detection exceeds lowest HHSL
Detection exceeds ESL
Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR LEACHATE VOCs (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
1,2-Dichloroethane	5	0.17	0.6	2.2	100
1,4-Dichlorobenzene	75	0.48	3	2.6	15
Benzene	5	0.46	1	1.6	46
Carbon disulfide	--	81	60	1200	0.92
Chlorobenzene	100	7.8	5	410	1.3
Methylene Chloride	5	11	5	760	98.1
Toluene	1000	110	5	19000	2
Trichloroethene	5	0.28	5	1.2	21
Vinyl chloride	2	0.019	2	0.15	--

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated; J+: biased high.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

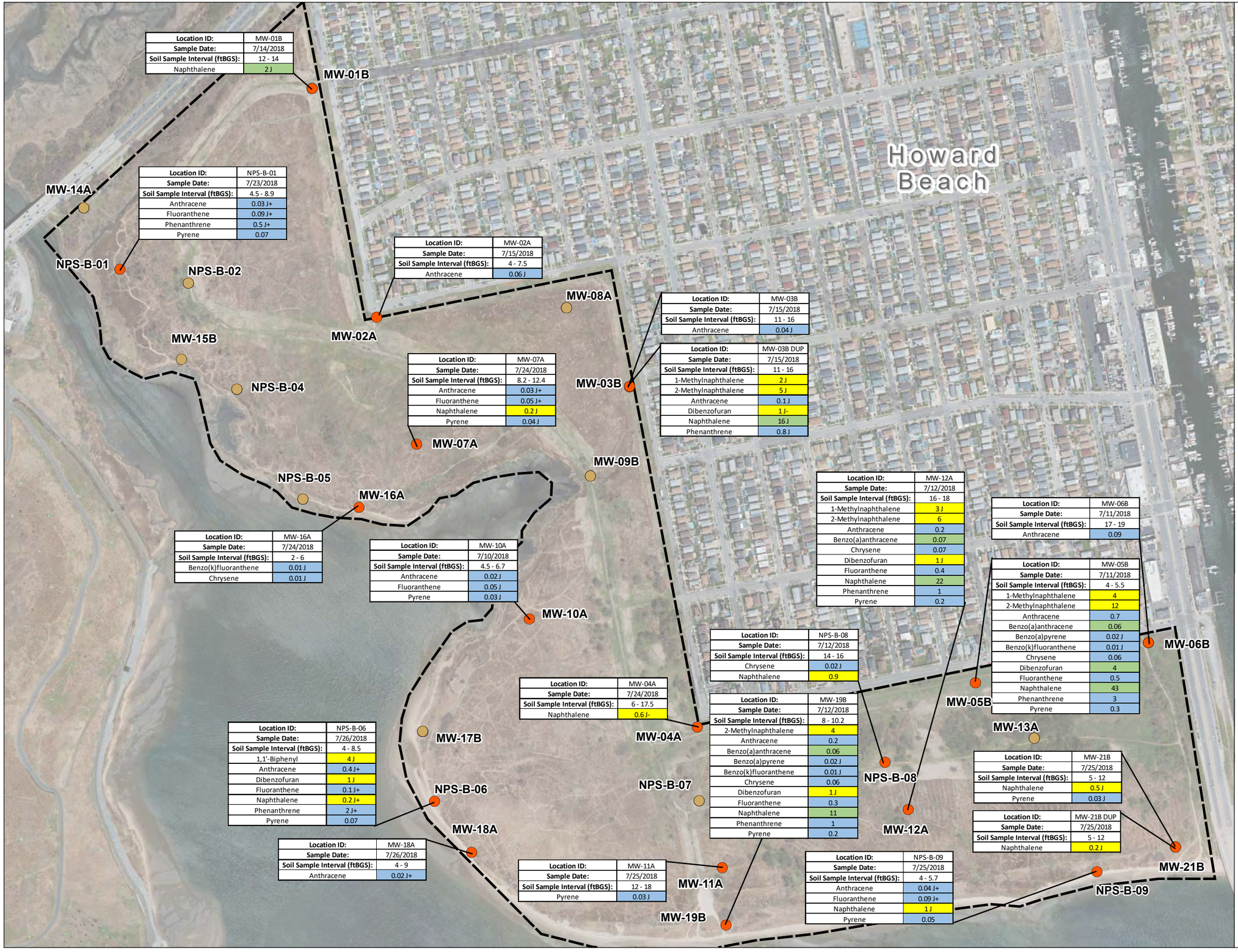
Figure 3.3: Exceedances of VOCs in Leachate
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/09/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.3 - Exceedances of VOCs in Leachate.mxd



Location ID:	MW-01B
Sample Date:	7/14/2018
Soil Sample Interval (ftBGS):	12 - 14
Naphthalene	2 J

Location ID:	NPS-B-01
Sample Date:	7/23/2018
Soil Sample Interval (ftBGS):	4.5 - 8.9
Anthracene	0.03 J+
Fluoranthene	0.09 J+
Phenanthrene	0.5 J+
Pyrene	0.07

Location ID:	MW-02A
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	4 - 7.5
Anthracene	0.06 J

Location ID:	MW-03B
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	11 - 16
Anthracene	0.04 J

Location ID:	MW-03B DUP
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	11 - 16
1-Methylnaphthalene	2 J
2-Methylnaphthalene	5 J
Anthracene	0.1 J
Dibenzofuran	1 J-
Naphthalene	16 J
Phenanthrene	0.8 J

Location ID:	MW-07A
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	8.2 - 12.4
Anthracene	0.03 J+
Fluoranthene	0.05 J+
Naphthalene	0.2 J
Pyrene	0.04 J

Location ID:	MW-12A
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	16 - 18
1-Methylnaphthalene	3 J
2-Methylnaphthalene	6
Anthracene	0.2
Benzo(a)anthracene	0.07
Chrysene	0.07
Dibenzofuran	1 J
Fluoranthene	0.4
Naphthalene	22
Phenanthrene	1
Pyrene	0.2

Location ID:	MW-06B
Sample Date:	7/11/2018
Soil Sample Interval (ftBGS):	17 - 19
Anthracene	0.09

Location ID:	MW-05B
Sample Date:	7/11/2018
Soil Sample Interval (ftBGS):	4 - 5.5
1-Methylnaphthalene	4
2-Methylnaphthalene	12
Anthracene	0.7
Benzo(a)anthracene	0.06
Benzo(a)pyrene	0.02 J
Benzo(k)fluoranthene	0.01 J
Chrysene	0.06
Dibenzofuran	4
Fluoranthene	0.5
Naphthalene	43
Phenanthrene	3
Pyrene	0.3

Location ID:	MW-16A
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	2 - 6
Benzo(k)fluoranthene	0.01 J
Chrysene	0.01 J

Location ID:	MW-10A
Sample Date:	7/10/2018
Soil Sample Interval (ftBGS):	4.5 - 6.7
Anthracene	0.02 J
Fluoranthene	0.05 J
Pyrene	0.03 J

Location ID:	NPS-B-08
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	14 - 16
Chrysene	0.02 J
Naphthalene	0.9

Location ID:	MW-04A
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	6 - 17.5
Naphthalene	0.6 J-

Location ID:	MW-19B
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	8 - 10.2
2-Methylnaphthalene	4
Anthracene	0.2
Benzo(a)anthracene	0.06
Benzo(a)pyrene	0.02 J
Benzo(k)fluoranthene	0.01 J
Chrysene	0.06
Dibenzofuran	1 J
Fluoranthene	0.3
Naphthalene	11
Phenanthrene	1
Pyrene	0.2

Location ID:	NPS-B-06
Sample Date:	7/26/2018
Soil Sample Interval (ftBGS):	4 - 8.5
1,1'-Biphenyl	4 J
Anthracene	0.4 J+
Dibenzofuran	1 J
Fluoranthene	0.1 J+
Naphthalene	0.2 J+
Phenanthrene	2 J+
Pyrene	0.07

Location ID:	MW-21B
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	5 - 12
Naphthalene	0.5 J
Pyrene	0.03 J

Location ID:	MW-21B DUP
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	5 - 12
Naphthalene	0.2 J

Location ID:	MW-18A
Sample Date:	7/26/2018
Soil Sample Interval (ftBGS):	4 - 9
Anthracene	0.02 J+

Location ID:	MW-11A
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	12 - 18
Pyrene	0.03 J

Location ID:	NPS-B-09
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	4 - 5.7
Anthracene	0.04 J+
Fluoranthene	0.09 J+
Naphthalene	1 J
Pyrene	0.05

Legend

- Soil/Fill Sample - At Least One Screening Level Exceedance
- Soil/Fill Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR LEACHATE SVOCs (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
1,1'-Biphenyl	--	0.083	5	33	--
1-Methylnaphthalene	--	1.1	--	--	--
2-Methylnaphthalene	--	3.6	--	--	330
Anthracene	--	180	--	--	0.012
Benzo(a)anthracene	--	0.03	--	--	0.018
Benzo(a)pyrene	0.2	0.025	--	--	0.014
Benzo(k)fluoranthene	--	2.5	--	--	0.0041
Chrysene	--	25	--	--	0.0018
Dibenzofuran	--	0.79	--	--	3.7
Fluoranthene	--	80	--	--	0.04
Naphthalene	--	0.17	--	4.6	1.1
Phenanthrene	--	--	--	--	0.4
Pyrene	--	12	--	--	0.025

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated; J+: biased high; J-: biased low.

TN

0 250 500 750 1,000
Feet

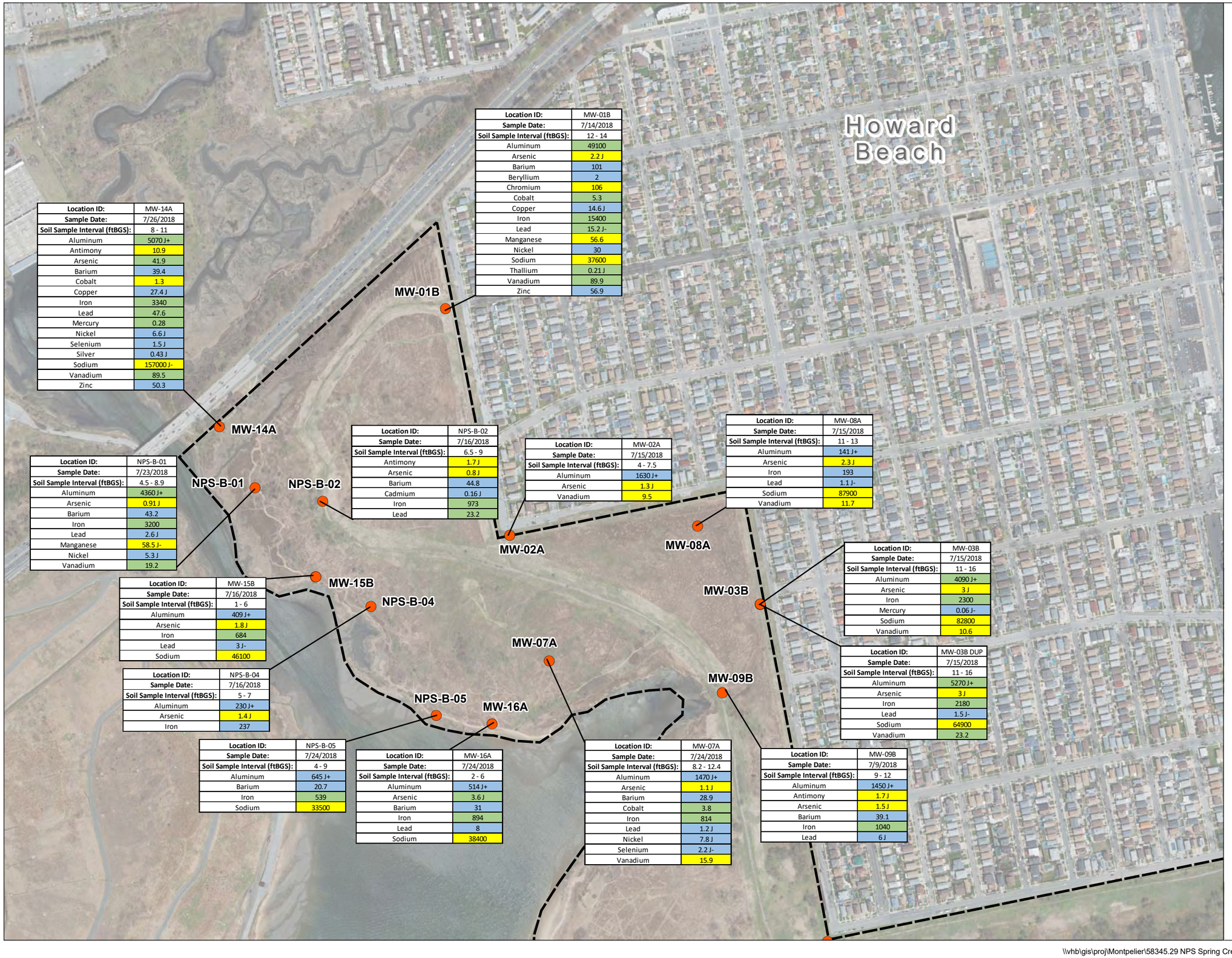
Figure 3.4: Exceedances of SVOCs in Leachate
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.4 - Exceedances of SVOCs in Leachate.mxd



Location ID:	MW-14A
Sample Date:	7/26/2018
Soil Sample Interval (ftBGS):	8 - 11
Aluminum	5070 J+
Antimony	10.9
Arsenic	41.9
Barium	39.4
Cobalt	1.3
Copper	27.4 J
Iron	3340
Lead	47.6
Mercury	0.28
Nickel	6.6 J
Selenium	1.5 J
Silver	0.43 J
Sodium	157000 J-
Vanadium	89.5
Zinc	50.3

Location ID:	MW-01B
Sample Date:	7/14/2018
Soil Sample Interval (ftBGS):	12 - 14
Aluminum	49100
Arsenic	2.2 J
Barium	101
Beryllium	2
Chromium	106
Cobalt	5.3
Copper	14.6 J
Iron	15400
Lead	15.2 J-
Manganese	56.6
Nickel	30
Sodium	37600
Thallium	0.21 J
Vanadium	89.9
Zinc	56.9

Location ID:	NPS-B-02
Sample Date:	7/16/2018
Soil Sample Interval (ftBGS):	6.5 - 9
Antimony	1.7 J
Arsenic	0.8 J
Barium	44.8
Cadmium	0.16 J
Iron	973
Lead	23.2

Location ID:	MW-02A
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	4 - 7.5
Aluminum	1630 J+
Arsenic	1.3 J
Vanadium	9.5

Location ID:	MW-08A
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	11 - 13
Aluminum	141 J+
Arsenic	2.3 J
Iron	193
Lead	1.1 J-
Sodium	87900
Vanadium	11.7

Location ID:	NPS-B-01
Sample Date:	7/23/2018
Soil Sample Interval (ftBGS):	4.5 - 8.9
Aluminum	4360 J+
Arsenic	0.91 J
Barium	43.2
Iron	3200
Lead	2.6 J
Manganese	58.5 J-
Nickel	5.3 J
Vanadium	19.2

Location ID:	MW-15B
Sample Date:	7/16/2018
Soil Sample Interval (ftBGS):	1 - 6
Aluminum	409 J+
Arsenic	1.8 J
Iron	684
Lead	3 J-
Sodium	46100

Location ID:	MW-03B
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	11 - 16
Aluminum	4090 J+
Arsenic	3 J
Iron	2300
Mercury	0.06 J-
Sodium	82800
Vanadium	10.6

Location ID:	MW-03B DUP
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	11 - 16
Aluminum	5270 J+
Arsenic	3 J
Iron	2180
Lead	1.5 J-
Sodium	64900
Vanadium	23.2

Location ID:	NPS-B-04
Sample Date:	7/16/2018
Soil Sample Interval (ftBGS):	5 - 7
Aluminum	230 J+
Arsenic	1.4 J
Iron	237

Location ID:	NPS-B-05
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	4 - 9
Aluminum	645 J+
Barium	20.7
Iron	539
Sodium	33500

Location ID:	MW-16A
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	2 - 6
Aluminum	514 J+
Arsenic	3.6 J
Barium	31
Iron	894
Lead	8
Sodium	38400

Location ID:	MW-07A
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	8.2 - 12.4
Aluminum	1470 J+
Arsenic	1.1 J
Barium	28.9
Cobalt	3.8
Iron	814
Lead	1.2 J
Nickel	7.8 J
Selenium	2.2 J-
Vanadium	15.9

Location ID:	MW-09B
Sample Date:	7/9/2018
Soil Sample Interval (ftBGS):	9 - 12
Aluminum	1450 J+
Antimony	1.7 J
Arsenic	1.5 J
Barium	39.1
Iron	1040
Lead	6 J

Legend

- Soil/Fill Sample - At Least One Screening Level Exceedance
- Soil/Fill Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR LEACHATE METALS (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
Aluminum	--	2000	--	--	5
Antimony	6	0.78	3	--	30
Arsenic	10	0.052	25	--	3.1
Barium	2000	380	1000	--	3.9
Beryllium	4	2.5	--	--	0.66
Cadmium	5	0.92	5	--	0.07
Chromium	100	--	50	--	--
Cobalt	--	0.6	--	--	3
Copper	1300	80	200	--	0.23
Iron	--	1400	300	--	158
Lead	15	15	25	--	0.92
Manganese	--	43	300	--	112
Mercury	2	0.063	0.7	0.89	0.026
Nickel	--	39	100	--	5
Selenium	50	10	10	--	1
Silver	--	9.4	50	--	0.067
Sodium	--	--	20000	--	680000
Thallium	2	0.02	--	--	0.03
Vanadium	--	8.6	--	--	19
Zinc	--	600	--	--	30

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated. J+: biased high; J-: biased low.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

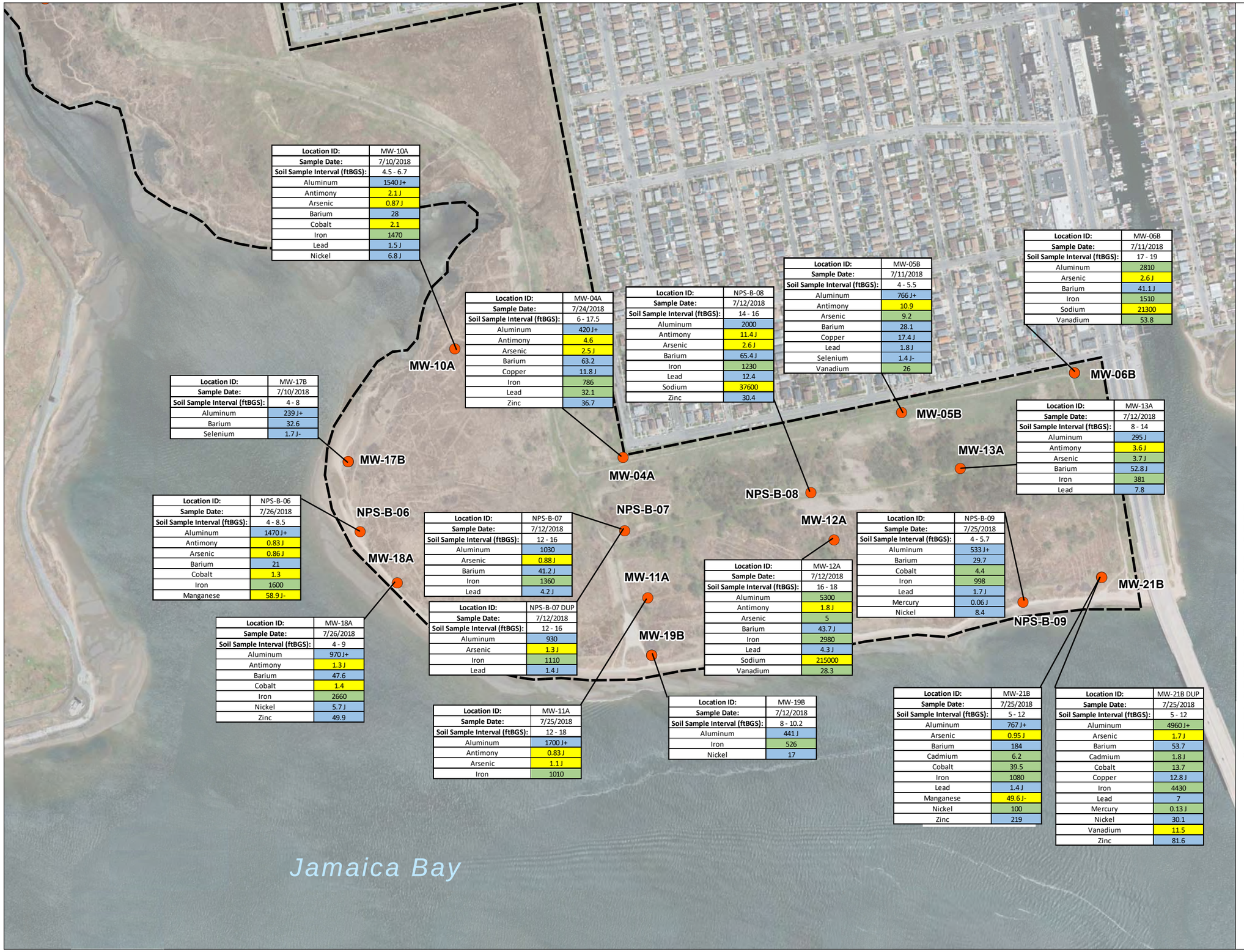
Figure 3.5a: Exceedances of Metals in SPLP Leachate (North)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.5a - Exceedances of Metals in Leachate (North).mxd



Location ID:	MW-10A
Sample Date:	7/10/2018
Soil Sample Interval (ftBGS):	4.5 - 6.7
Aluminum	1540 J+
Antimony	2.1 J
Arsenic	0.87 J
Barium	28
Cobalt	2.1
Iron	1470
Lead	1.5 J
Nickel	6.8 J

Location ID:	MW-17B
Sample Date:	7/10/2018
Soil Sample Interval (ftBGS):	4 - 8
Aluminum	239 J+
Barium	32.6
Selenium	1.7 J-

Location ID:	NPS-B-06
Sample Date:	7/26/2018
Soil Sample Interval (ftBGS):	4 - 8.5
Aluminum	1470 J+
Antimony	0.83 J
Arsenic	0.86 J
Barium	21
Cobalt	1.3
Iron	1600
Manganese	58.9 J-

Location ID:	MW-18A
Sample Date:	7/26/2018
Soil Sample Interval (ftBGS):	4 - 9
Aluminum	970 J+
Antimony	1.3 J
Barium	47.6
Cobalt	1.4
Iron	2660
Nickel	5.7 J
Zinc	49.9

Location ID:	MW-04A
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	6 - 17.5
Aluminum	420 J+
Antimony	4.6
Arsenic	2.5 J
Barium	63.2
Copper	11.8 J
Iron	786
Lead	32.1
Zinc	36.7

Location ID:	NPS-B-07
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	12 - 16
Aluminum	1030
Arsenic	0.88 J
Barium	41.2 J
Iron	1360
Lead	4.2 J

Location ID:	NPS-B-07 DUP
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	12 - 16
Aluminum	930
Arsenic	1.3 J
Iron	1110
Lead	1.4 J

Location ID:	MW-11A
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	12 - 18
Aluminum	1700 J+
Antimony	0.83 J
Arsenic	1.1 J
Iron	1010

Location ID:	NPS-B-08
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	14 - 16
Aluminum	2000
Antimony	11.4 J
Arsenic	2.6 J
Barium	65.4 J
Iron	1230
Lead	12.4
Sodium	37600
Zinc	30.4

Location ID:	MW-19B
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	8 - 10.2
Aluminum	441 J
Iron	526
Nickel	17

Location ID:	MW-05B
Sample Date:	7/11/2018
Soil Sample Interval (ftBGS):	4 - 5.5
Aluminum	766 J+
Antimony	10.9
Arsenic	9.2
Barium	28.1
Copper	17.4 J
Lead	1.8 J
Selenium	1.4 J-
Vanadium	26

Location ID:	MW-12A
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	16 - 18
Aluminum	5300
Antimony	1.8 J
Arsenic	5
Barium	43.7 J
Iron	2980
Lead	4.3 J
Sodium	215000
Vanadium	28.3

Location ID:	NPS-B-09
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	4 - 5.7
Aluminum	533 J+
Barium	29.7
Cobalt	4.4
Iron	998
Lead	1.7 J
Mercury	0.06 J
Nickel	8.4

Location ID:	MW-06B
Sample Date:	7/11/2018
Soil Sample Interval (ftBGS):	17 - 19
Aluminum	2810
Arsenic	2.6 J
Barium	41.1 J
Iron	1510
Sodium	21300
Vanadium	53.8

Location ID:	MW-13A
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	8 - 14
Aluminum	295 J
Antimony	3.6 J
Arsenic	3.7 J
Barium	52.8 J
Iron	381
Lead	7.8

Location ID:	MW-21B
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	5 - 12
Aluminum	767 J+
Arsenic	0.95 J
Barium	184
Cadmium	6.2
Cobalt	39.5
Iron	1080
Lead	1.4 J
Manganese	49.6 J-
Nickel	100
Zinc	219

Location ID:	MW-21B DUP
Sample Date:	7/25/2018
Soil Sample Interval (ftBGS):	5 - 12
Aluminum	4960 J+
Arsenic	1.7 J
Barium	53.7
Cadmium	1.8 J
Cobalt	13.7
Copper	12.8 J
Iron	4430
Lead	7
Mercury	0.13 J
Nickel	30.1
Vanadium	11.5
Zinc	81.6

Legend

- Soil/Fill Sample - At Least One Screening Level Exceedance
- Soil/Fill Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR LEACHATE METALS (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
Aluminum	--	2000	--	--	5
Antimony	6	0.78	3	--	30
Arsenic	10	0.052	25	--	3.1
Barium	2000	380	1000	--	3.9
Beryllium	4	2.5	--	--	0.66
Cadmium	5	0.92	5	--	0.07
Chromium	100	--	50	--	--
Cobalt	--	0.6	--	--	3
Copper	1300	80	200	--	0.23
Iron	--	1400	300	--	158
Lead	15	15	25	--	0.92
Manganese	--	43	300	--	112
Mercury	2	0.063	0.7	0.89	0.026
Nickel	--	39	100	--	5
Selenium	50	10	10	--	1
Silver	--	9.4	50	--	0.067
Sodium	--	--	20000	--	680000
Thallium	2	0.02	--	--	0.03
Vanadium	--	8.6	--	--	19
Zinc	--	600	--	--	30

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated. J+: biased high; J-: biased low.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.5b: Exceedances of Metals in SPLP Leachate (South)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.5b - Exceedances of Metals in Leachate (South).mxd

Location ID:	NPS-B-02
Sample Date:	7/16/2018
Soil Sample Interval (ftBGS):	6.5 - 9
Total PCBs (µg/L)	0.089 J-
Dioxins and Furans TEQ (pg/L)	5.84 J

Location ID:	MW-02A
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	4 - 7.5
Total PCBs (µg/L)	0.029

Location ID:	MW-03B DUP
Sample Date:	7/15/2018
Soil Sample Interval (ftBGS):	11 - 16
Dioxins and Furans TEQ (pg/L)	0.14 J

Location ID:	MW-15B
Sample Date:	7/16/2018
Soil Sample Interval (ftBGS):	1 - 6
Total PCBs (µg/L)	0.016

Location ID:	MW-16A
Sample Date:	7/24/2018
Soil Sample Interval (ftBGS):	2 - 6
Total PCBs (µg/L)	0.015
Dioxins and Furans TEQ (pg/L)	2.6 J

Location ID:	MW-05B
Sample Date:	7/11/2018
Soil Sample Interval (ftBGS):	4 - 5.5
cis-Chlordane (µg/L)	0.0092 J
gamma-Chlordane (µg/L)	0.011 J+
4,4-DDT (µg/L)	0.0057 J

Location ID:	MW-06B
Sample Date:	7/11/2018
Soil Sample Interval (ftBGS):	17 - 19
Dioxins and Furans TEQ (pg/L)	1.3 J

Location ID:	NPS-B-08
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	14 - 16
Total PCBs (µg/L)	0.039 JN
Dioxins and Furans TEQ (pg/L)	5.5 J

Location ID:	NPS-B-07
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	12 - 16
Dioxins and Furans TEQ (pg/L)	0.34 J

Location ID:	MW-13A
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	8 - 14
Dioxins and Furans TEQ (pg/L)	0.196 J

Location ID:	MW-12A
Sample Date:	7/12/2018
Soil Sample Interval (ftBGS):	16 - 18
Total PCBs (µg/L)	0.022
Dioxins and Furans TEQ (pg/L)	0.523 J

Legend

- Soil/Fill Sample - At Least One Screening Level Exceedance
- Soil/Fill Sample - No Screening Level Exceedances
- Approximate Site Boundary

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR LEACHATE PESTICIDES, PCBs, AND DIOXINS AND FURANS

Analyte	Units	MCL	TW	GA	ESV
4,4'-DDT	µg/L	--	0.23	0.2	0.001
cis-Chlordane	µg/L	--	--	--	0.0043
gamma-Chlordane	µg/L	--	--	--	0.0043
Total PCBs	µg/L	0.5	--	0.09	0.014
Dioxins and Furans TEQ	pg/L	--	0.12	0.7	2

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated; J+: biased high; J-: biased low.
TEQ: Toxic Equivalent.
Dioxins and Furans concentrations via TEQ.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

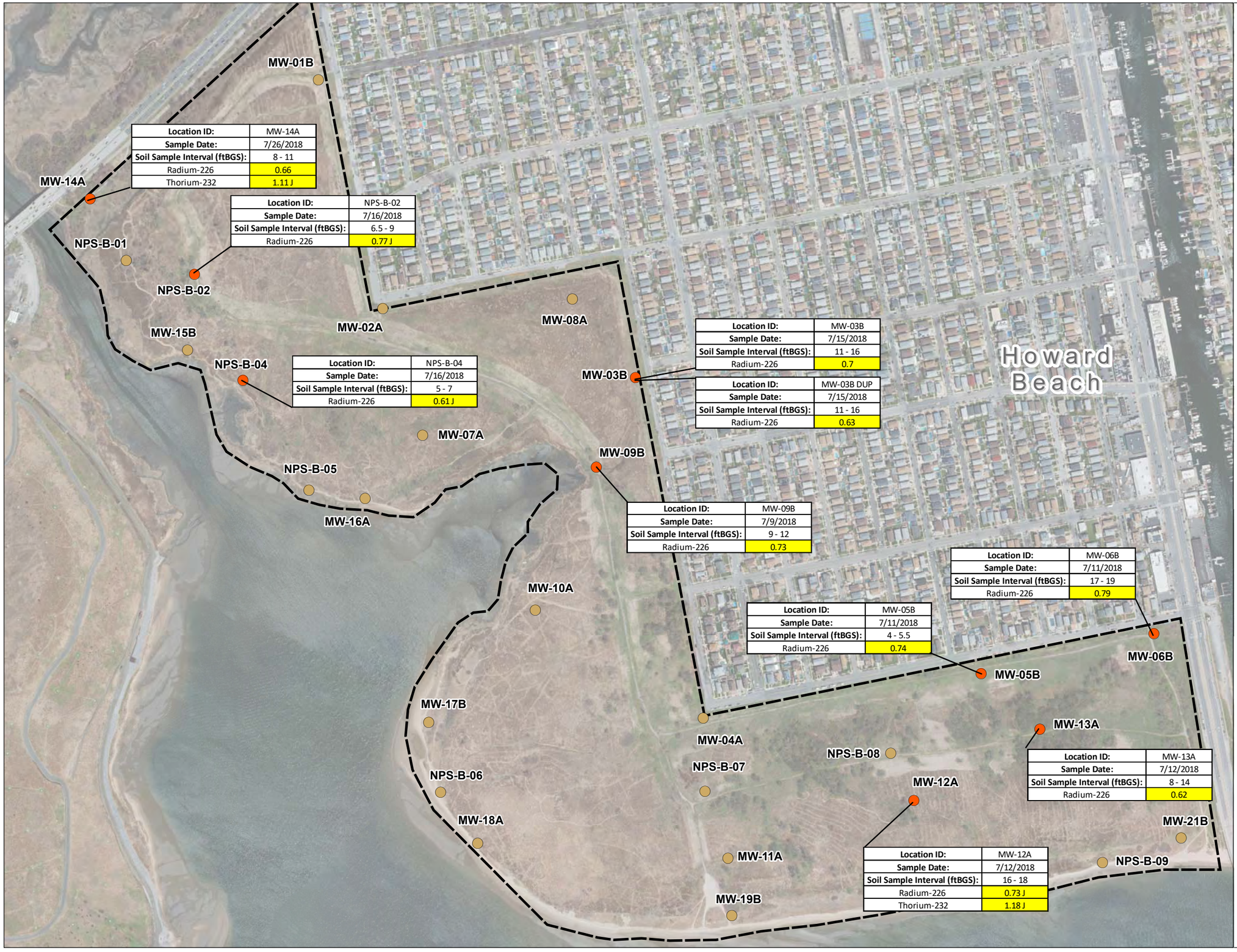
0 240 480 720 960
Feet

Figure 3.6: Exceedances of Pesticides, PCBs, and Dioxins and Furans in SPLP Leachate
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.6 - Exceedances of Pesticides, PCBs, and Dioxins and Furans in Leachate.mxd



Legend

- Soil/Fill Sample - At Least One Screening Level Exceedance
- Soil/Fill Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in pCi/g.

	Detection exceeds HHSL
	Detection exceeds ESL
	Detection exceeds both HHSL and ESL

SCREENING LEVELS FOR RADIOCHEMISTRY IN SOIL (pCi/g)

Analyte	NUREG	Terrestrial ESV
Radium-226	0.6	1.5
Thorium-232 (via Radium-228)	1.1	6.2

Notes:
NUREG: US Nuclear Regulatory Commission Regulation.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated; J+: biased high.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.7: Exceedances of Radionuclides in Soil/Fill
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.7 - Exceedances of Radionuclides in SoilFill.mxd

Legend

- Shallow and Deep Monitoring Well Pair
- Shallow Monitoring Well
  Water Elevation in Shallow Wells (ft)
- Deep Monitoring Well
- Soil Boring
- Surface Water Sample
- Staff Gauge
- Approximate Site Boundary
- Water Level Contour (1988 NAVD ft)
- Inferred Water Level Contour (1988 NAVD ft)
- Inferred Groundwater Flow Direction
- Storm Sewer

Notes:
Groundwater elevation data were collected 28 July 2018. Groundwater elevation contours were prepared using linear interpolation of elevations between monitoring wells and adjusted to fit the site conceptual models (ground surface contours, storm sewer locations, coastal features). Other interpretations are possible.
Location data provided by Rogers Surveying (August 2018).
Basemap from ESRI.

TN

0 250 500 750 1,000
Feet

Figure 3.8a: Groundwater Elevation Contours (Shallow, High Tide)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 11/27/2018
Reviewed by: BNH Date: 12/3/2018

Scale: 1 " = 500 ' Project: 3-0700-29

K:\3-0700-29 Spring Creek\GIS\MXDs\Figure 5-1a Groundwater Contours - High Tide Shallow.mxd

SW-04
SW-03
MW-01A
3.96
MW-01B
MW-14A
2.23
SW-05
NPS-B-03
NPS-B-01
NPS-B-02
MW-02A
4.33
MW-08A
3.95
MW-15A
2.10
MW-15B
NPS-B-04
MW-03A
4.53
MW-03B
MW-07A
3.85
MW-09A
1.95
MW-09B
Staff Gauge 1
0.72
SW-01
NPS-B-05
MW-16A
2.26
SW-02
MW-10A
2.55
MW-06B
MW-05B
MW-05A
3.95
Staff Gauge 2
-1.15
MW-17A
2.13
MW-17B
MW-04A
3.96
MW-13A
4.17
NPS-B-08
NPS-B-06
NPS-B-07
MW-12A
4.28
MW-18A
1.85
MW-21B
MW-21A
1.84
MW-11A
3.33
NPS-B-09
MW-20A
2.61
MW-19A
2.07
MW-19B
2

Legend

- Shallow and Deep Monitoring Well Pair
- Shallow Monitoring Well
  Water Elevation in Shallow Wells (ft)
- Deep Monitoring Well
- Soil Boring
- Surface Water Sample
- Staff Gauge
- Approximate Site Boundary
- Water Level Contour (1988 NAVD ft)
- Inferred Water Level Contour (1988 NAVD ft)
- Inferred Groundwater Flow Direction
- Storm Sewer

Notes:
Groundwater elevation data were collected 27 July 2018. Groundwater elevation contours were prepared using linear interpolation of elevations between monitoring wells and adjusted to fit the site conceptual models (ground surface contours, storm sewer locations, coastal features). Other interpretations are possible.
Location data provided by Rogers Surveying (August 2018).
Basemap from ESRI.

TN

0 250 500 750 1,000
Feet

Figure 3.8b: Groundwater Elevation Contours
(Shallow, Low Tide)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

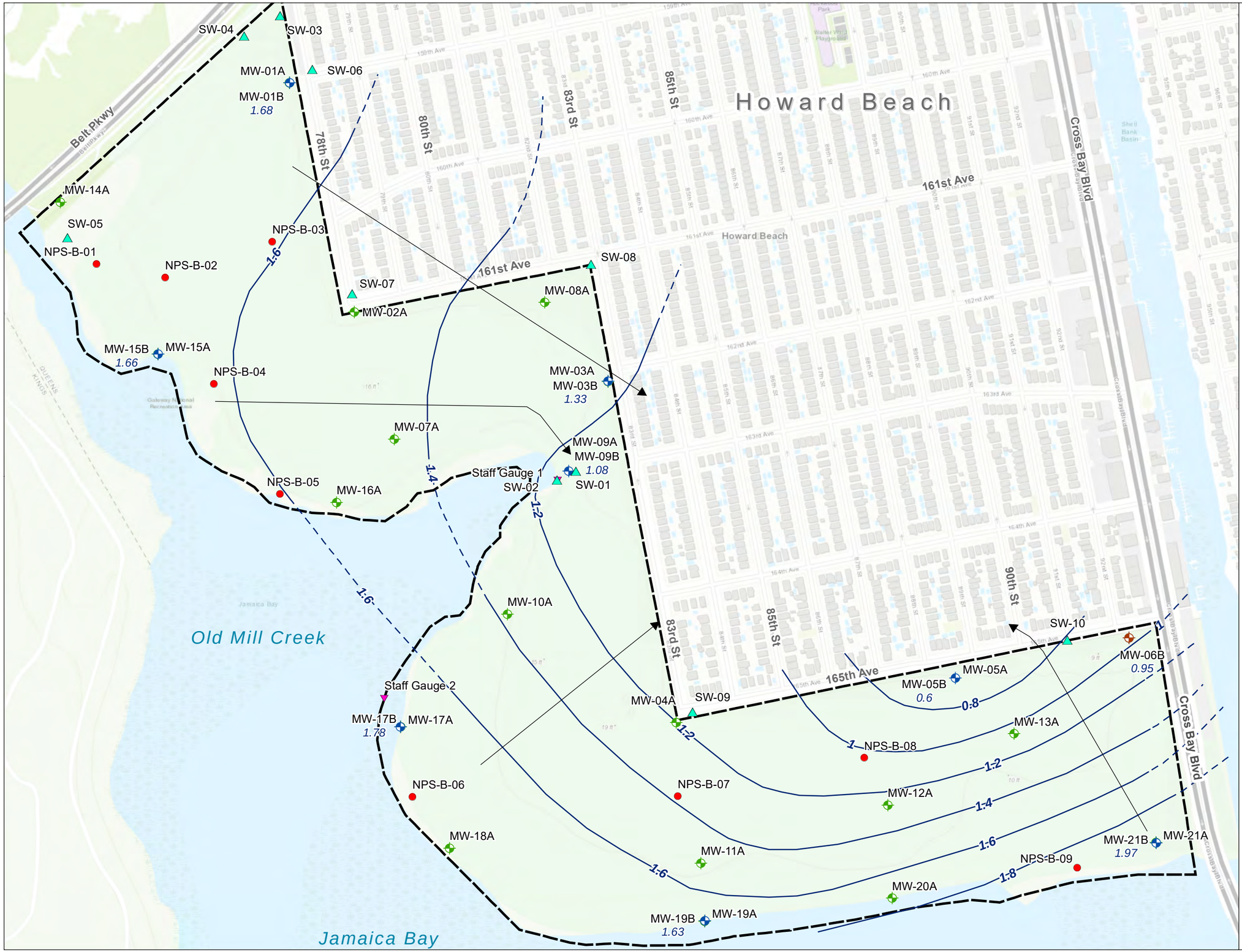
The Johnson Company

100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 11/27/2018
Reviewed by: BNH Date: 12/3/2018

Scale: 1 " = 500 ' Project: 3-0700-29

K:\3-0700-29 Spring Creek\GIS\MXDs\Figure 5-1b Groundwater Contours - Low Tide Shallow.mxd



Legend

- Shallow and Deep Monitoring Well Pair
  Water Elevation in Deep Wells (ft)
- Shallow Monitoring Well
- Deep Monitoring Well
- Soil Boring
- Surface Water Sample
- Staff Gauge
- Approximate Site Boundary
- Water Level Contour (1988 NAVD ft)
- Inferred Water Level Contour (1988 NAVD ft)
- Inferred Groundwater Flow Direction

Notes:
Groundwater elevation data were collected 28 July 2018. Groundwater elevation contours were prepared using linear interpolation of elevations between monitoring wells and adjusted to fit the site conceptual models (ground surface contours, storm sewer locations, coastal features). Other interpretations are possible.
Location data provided by Rogers Surveying (August 2018).
Basemap from ESRI.

TN

0 250 500 750 1,000
Feet

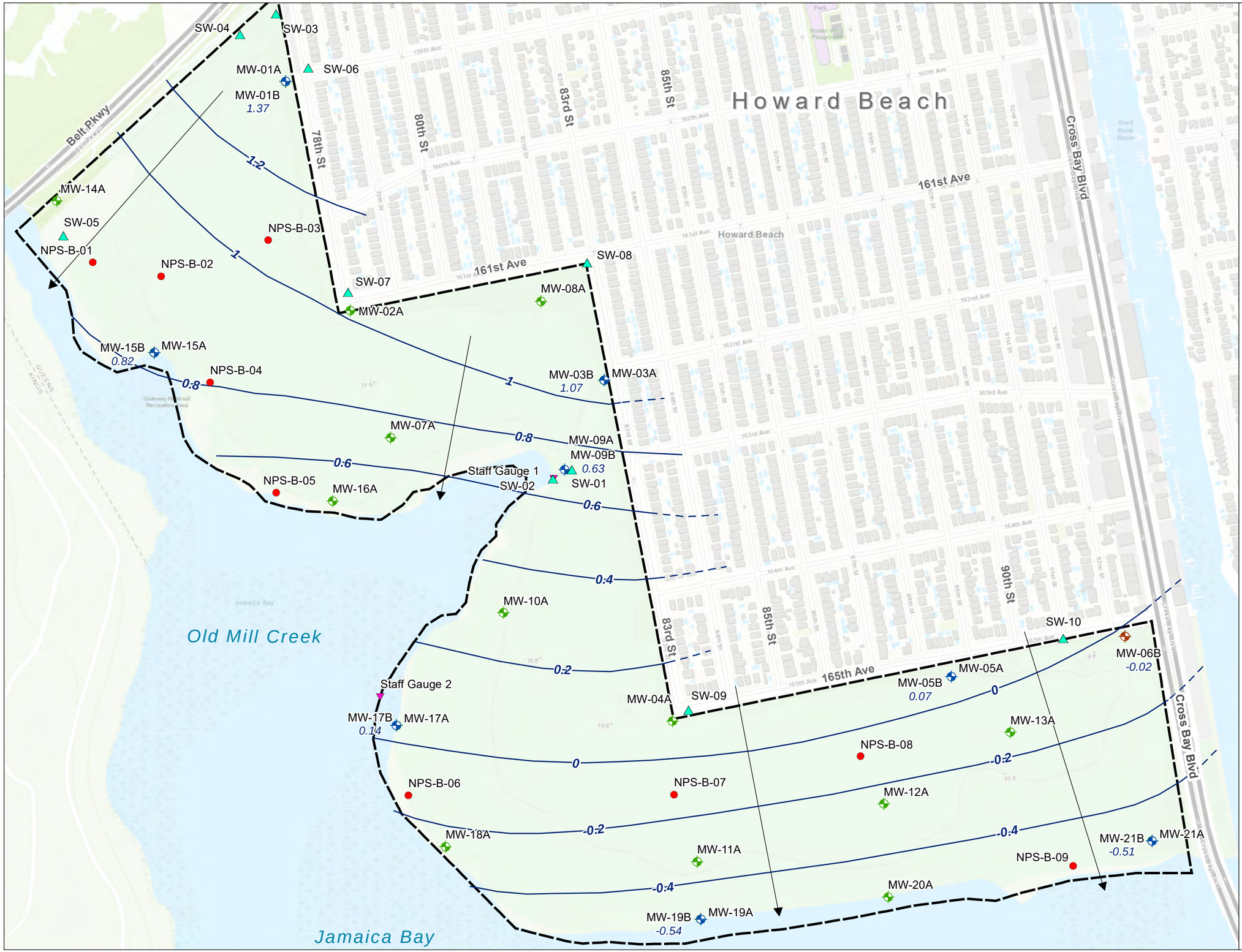
Figure 3.9a: Groundwater Elevation Contours (Deep, High Tide)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 11/27/2018
Reviewed by: BNH Date: 12/3/2018

Scale: 1 " = 500 ' Project: 3-0700-29

K:\3-0700-29 Spring Creek\GIS\MXDs\Figure 5-2a Groundwater Contours - High Tide Deep.mxd



Legend

- Shallow and Deep Monitoring Well Pair
  Water Elevation in Deep Wells (ft)
- Shallow Monitoring Well
- Deep Monitoring Well
- Soil Boring
- Surface Water Sample
- Staff Gauge
- Approximate Site Boundary
- Water Level Contour (1988 NAVD ft)
- Inferred Water Level Contour (1988 NAVD ft)
- Inferred Groundwater Flow Direction

Notes:
Groundwater elevation data were collected 28 July 2018. Groundwater elevation contours were prepared using linear interpolation of elevations between monitoring wells and adjusted to fit the site conceptual models (ground surface contours, storm sewer locations, coastal features). Other interpretations are possible.
Location data provided by Rogers Surveying (August 2018).
Basemap from ESRI.

TN

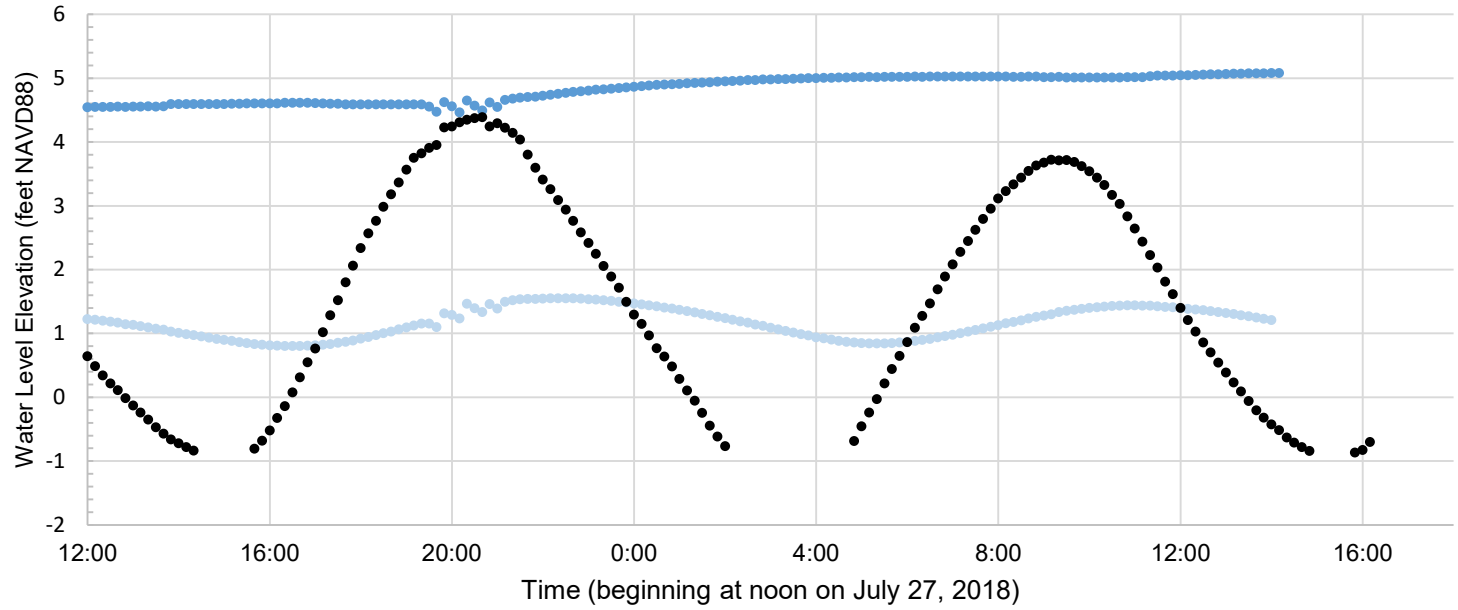
0 250 500 750 1,000
Feet

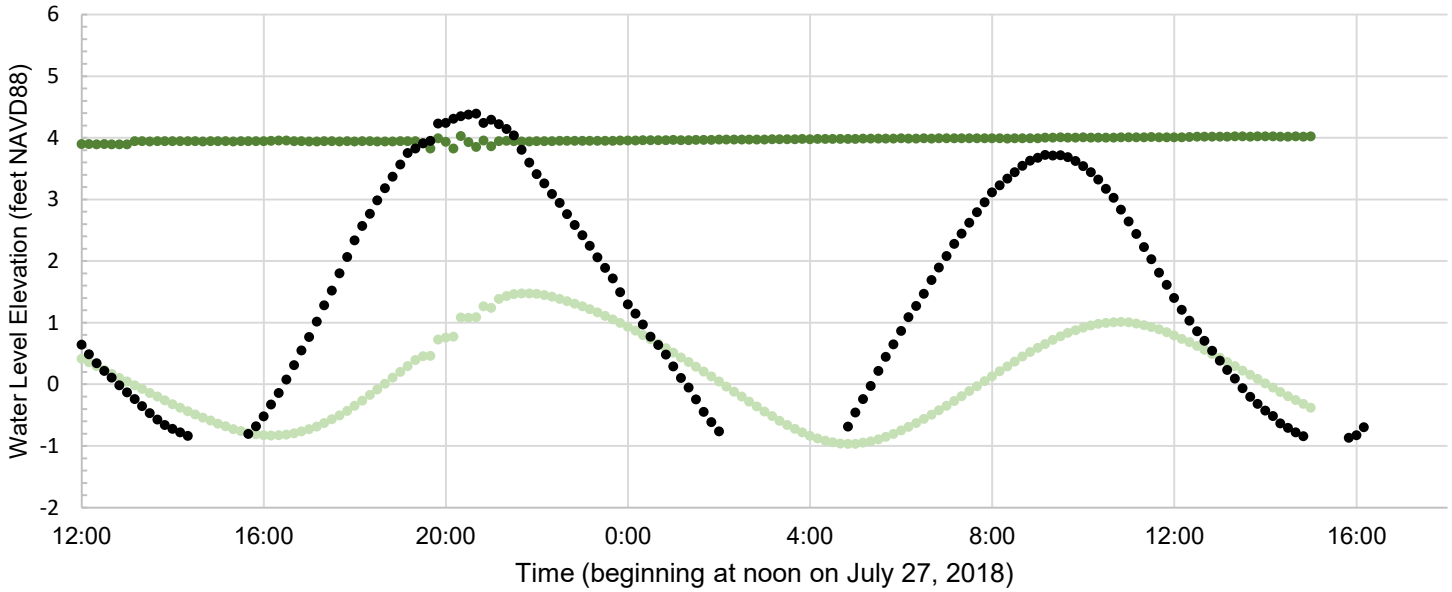
Figure 3.9b: Groundwater Elevation Contours (Deep, Low Tide)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 11/27/2018
Reviewed by: BNH Date: 12/3/2018
Scale: 1 " = 500 ' Project: 3-0700-29

K:\3-0700-29 Spring Creek\GIS\MXDs\Figure 5-2b Groundwater Contours - Low Tide Deep.mxd





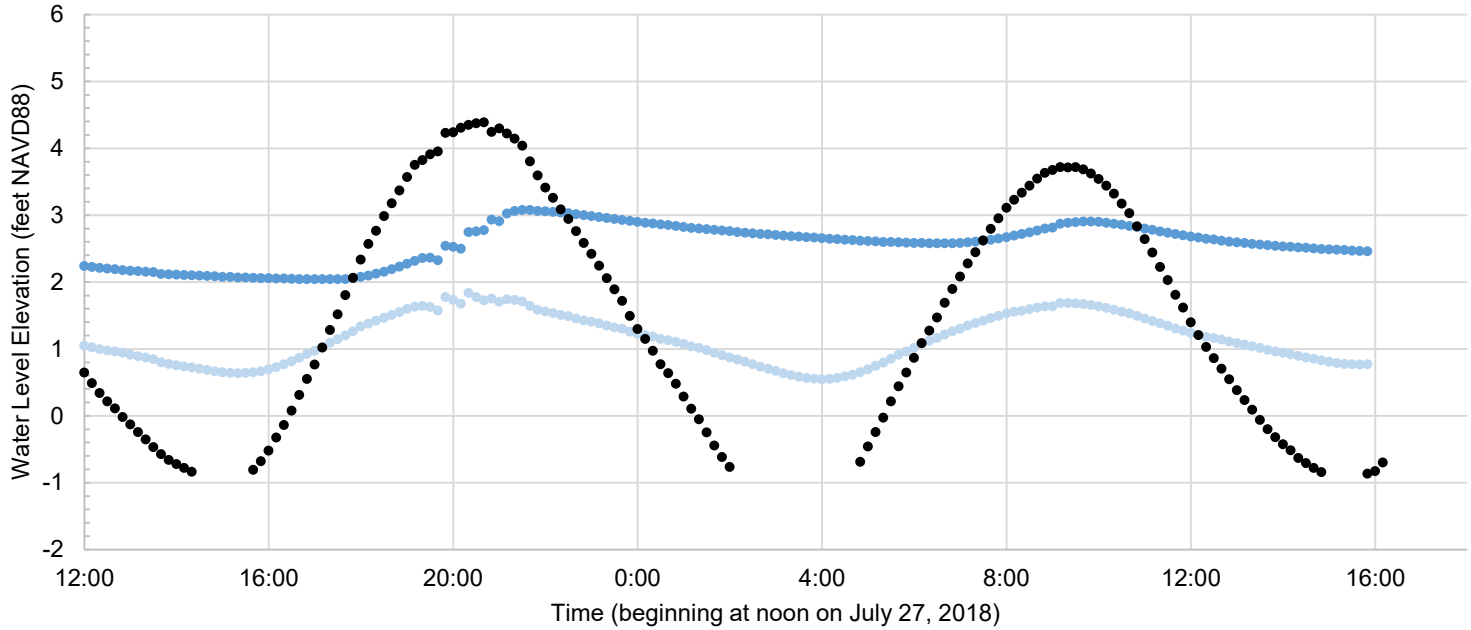
Notes:

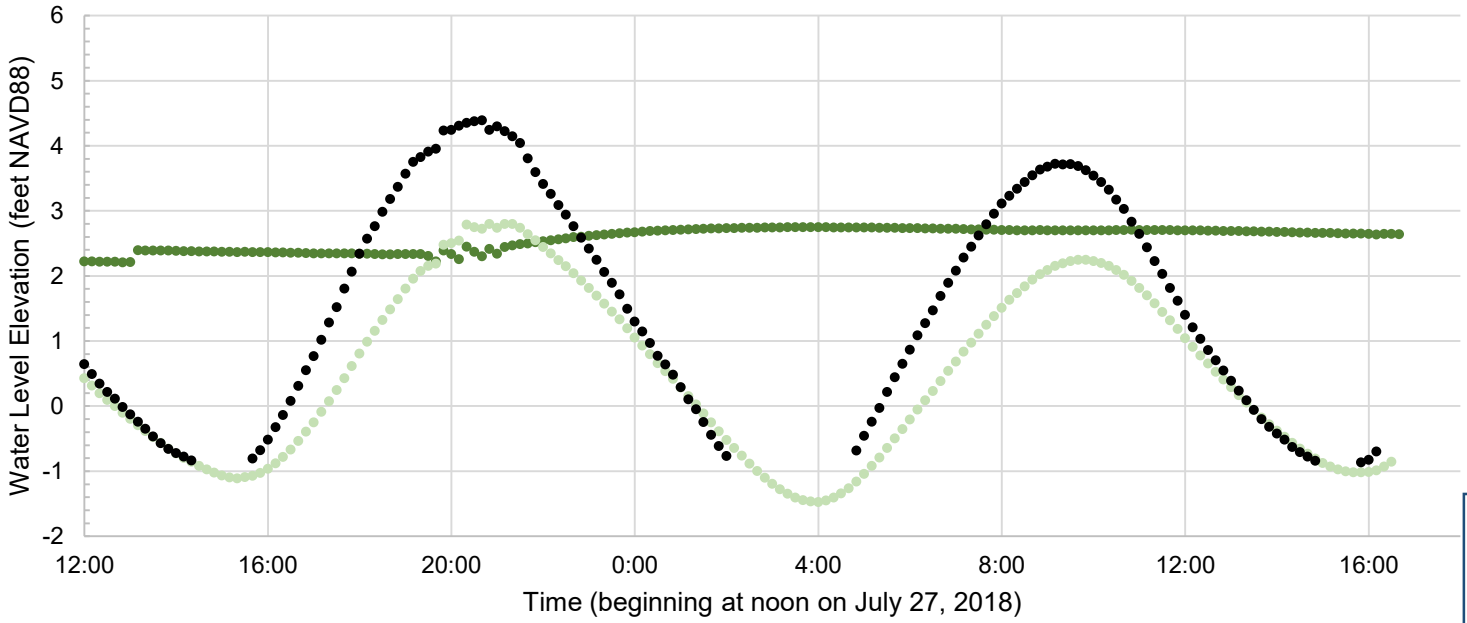
1) Spikes in the logger recording barometric pressure occurred between 7:40 PM - 9:00 PM on July 27; likely due to high wave action washing water over the data logger installed in SG-01. The resulting "chatter in water levels of approximately 0.2 feet has not been corrected.
2) The surface water logger installed at SG-02 was not sufficiently deep enough to measure the lowest levels of low tide, resulting in limited measures during the low tide cycles.

Figure 3.10a: Tidal Response in Inland Shallow and Deep Wells
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: NLS Date: 01/21/2019
Reviewed by: BNH Date: 01/21/2019
Project: 3-0700-29





Notes:

1) Spikes in the logger recording barometric pressure occurred between 7:40 PM - 9:00 PM on July 27; likely due to high wave action washing water over the data logger installed in SG-01. The resulting "chatter in water levels of approximately 0.2 feet has not been corrected.
2) The surface water logger installed at SG-02 was not sufficiently deep enough to measure the lowest levels of low tide, resulting in limited measures during the low tide cycles.

Figure 3.10b: Tidal Response in Coastal Shallow and Deep Wells
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: NLS Date: 01/21/2019
Reviewed by: BNH Date: 01/21/2019

Project: 3-0700-29

Location ID:	MW-01A	MW-01A
Sample Date:	7/19/2018	4/9/2019
Screen Interval (ftBGS):	3.25 - 12.75	3.25 - 12.75
1,4-Dichlorobenzene	2.3	2.1
Benzene	0.7	0.5
Chlorobenzene	13	13

Location ID:	MW-01A DUP
Sample Date:	7/19/2018
Screen Interval (ftBGS):	3.25 - 12.75
1,4-Dichlorobenzene	2.2
Benzene	0.6
Chlorobenzene	13

Location ID:	MW-02A	MW-02A
Sample Date:	7/19/2018	4/9/2019
Screen Interval (ftBGS):	5.25 - 9.75	5.25 - 9.75
1,4-Dichlorobenzene	1.2	0.7
Carbon disulfide	2.7 J	< 1
Chlorobenzene	6.8	3.6

Location ID:	MW-08A	MW-08A
Sample Date:	7/20/2018	4/11/2019
Screen Interval (ftBGS):	4.45 - 8.95	4.45 - 8.95
1,4-Dichlorobenzene	6.6	4.6
Benzene	3.1	1.7
Chlorobenzene	47	40

Location ID:	MW-07A	MW-07A
Sample Date:	7/31/2018	4/14/2019
Screen Interval (ftBGS):	7.35 - 11.85	7.35 - 11.85
1,4-Dichlorobenzene	1.6	1.4 J-
Chlorobenzene	17	24 J-

Location ID:	MW-16A	MW-16A
Sample Date:	8/1/2018	4/14/2019
Screen Interval (ftBGS):	3.05 - 7.55	3.05 - 7.55
1,4-Dichlorobenzene	0.3 J	0.9 J-
Chlorobenzene	< 0.5	4.2 J-
Vinyl chloride	< 0.5	1 J-

Location ID:	MW-10A	MW-10A
Sample Date:	7/23/2018	4/11/2019
Screen Interval (ftBGS):	3.95 - 13.45	3.95 - 13.45
1,4-Dichlorobenzene	2.3	1.2
Carbon disulfide	1.2	< 1
Chlorobenzene	13	8

Location ID:	MW-04A	MW-04A
Sample Date:	7/30/2018	4/9/2019
Screen Interval (ftBGS):	6.6 - 16.1	6.6 - 16.1
1,4-Dichlorobenzene	1.9	1.3
Chlorobenzene	4.3	2.2

Location ID:	MW-13A	MW-13A
Sample Date:	7/20/2018	4/14/2019
Screen Interval (ftBGS):	5.45 - 14.95	5.45 - 14.95
Vinyl chloride	< 0.5	0.1 J

Location ID:	MW-12A	MW-12A
Sample Date:	7/20/2018	4/15/2019
Screen Interval (ftBGS):	7.55 - 17.05	7.55 - 17.05
1,4-Dichlorobenzene	11	11
Benzene	2.7	2 J
Chlorobenzene	97	62

Location ID:	MW-11A	MW-11A
Sample Date:	7/31/2018	4/10/2019
Screen Interval (ftBGS):	9.25 - 18.75	9.25 - 18.75
Chlorobenzene	5.3	6.3

Location ID:	MW-11A DUP
Sample Date:	7/31/2018
Screen Interval (ftBGS):	9.25 - 18.75
Chlorobenzene	5.3

Location ID:	MW-19A	MW-19A
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	4.35 - 13.85	4.35 - 13.85
1,4-Dichlorobenzene	< 0.5	1
Chlorobenzene	< 0.5	4.7

Location ID:	MW-20A	MW-20A
Sample Date:	7/31/2018	4/16/2019
Screen Interval (ftBGS):	3.65 - 13.15	3.65 - 13.15
1,4-Dichlorobenzene	0.8	0.8
Chlorobenzene	2.7	2.5

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER VOCs (µg/L)					
Analyte	MCL	TW	GA	VISL	ESV
1,4-Dichlorobenzene	75	0.48	3	2.6	15
Benzene	5	0.46	1	1.6	46
Carbon disulfide	--	81	60	1200	0.92
Chlorobenzene	100	7.8	5	410	1.3
Vinyl chloride	2	0.019	2	0.15	--

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.11a: Exceedances of VOCs in Shallow Groundwater
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.11a - Exceedances f VOCs in Shallow Groundwater.mxd

Location ID:	MW-15B	MW-15B
Sample Date:	7/23/2018	4/14/2019
Screen Interval (ftBGS):	14.35 - 18.85	14.35 - 18.85
1,4-Dichlorobenzene	0.5 J	0.4 J

Location ID:	MW-03B	MW-03B
Sample Date:	7/18/2018	4/9/2019
Screen Interval (ftBGS):	17.2 - 21.7	17.2 - 21.7
Carbon disulfide	3.2	0.1 J

Location ID:	MW-21B	MW-21B
Sample Date:	7/31/2018	4/13/2019
Screen Interval (ftBGS):	19.25 - 23.75	19.25 - 23.75
Chloroform	0.5 J	< 0.5

Location ID:	MW-19B	MW-19B
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	20.25 - 24.75	20.25 - 24.75
1,4-Dichlorobenzene	0.9	< 0.5 J
Carbon disulfide	< 1	1 J-
Chlorobenzene	4.5	< 0.5 J

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER VOCs (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
1,4-Dichlorobenzene	75	0.48	3	2.6	15
Carbon disulfide	--	81	60	1200	0.92
Chlorobenzene	100	7.8	5	410	1.3
Chloroform	80	0.22	7	0.81	1.8

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

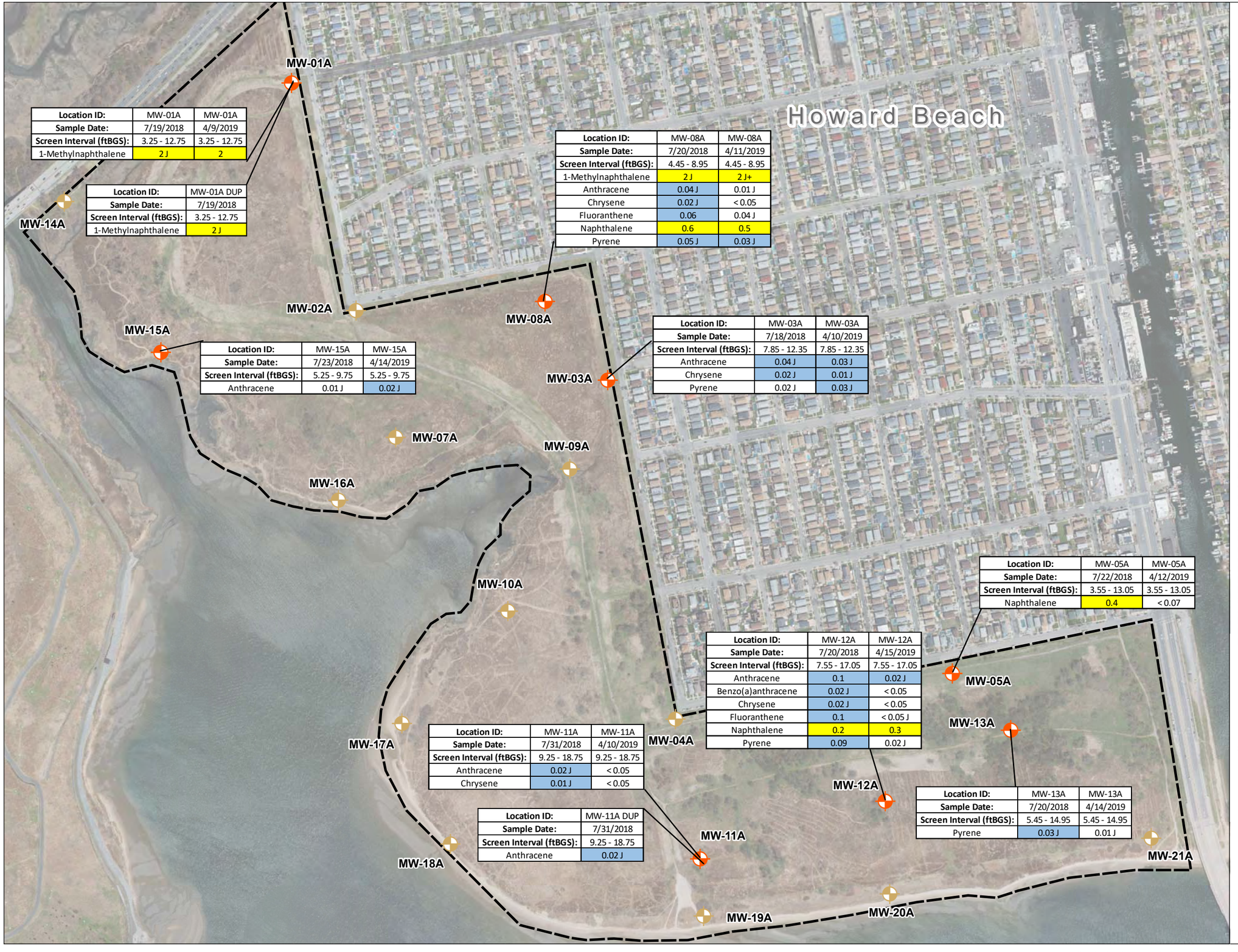
0 250 500 750 1,000
Feet

Figure 3.11b: Exceedances of VOCs in Deep Groundwater
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.11b - Exceedances of VOCs in Deep Groundwater.mxd



Location ID:	MW-01A	MW-01A
Sample Date:	7/19/2018	4/9/2019
Screen Interval (ftBGS):	3.25 - 12.75	3.25 - 12.75
1-Methylnaphthalene	2 J	2

Location ID:	MW-01A DUP
Sample Date:	7/19/2018
Screen Interval (ftBGS):	3.25 - 12.75
1-Methylnaphthalene	2 J

Location ID:	MW-08A	MW-08A
Sample Date:	7/20/2018	4/11/2019
Screen Interval (ftBGS):	4.45 - 8.95	4.45 - 8.95
1-Methylnaphthalene	2 J	2 J+
Anthracene	0.04 J	0.01 J
Chrysene	0.02 J	< 0.05
Fluoranthene	0.06	0.04 J
Naphthalene	0.6	0.5
Pyrene	0.05 J	0.03 J

Location ID:	MW-15A	MW-15A
Sample Date:	7/23/2018	4/14/2019
Screen Interval (ftBGS):	5.25 - 9.75	5.25 - 9.75
Anthracene	0.01 J	0.02 J

Location ID:	MW-03A	MW-03A
Sample Date:	7/18/2018	4/10/2019
Screen Interval (ftBGS):	7.85 - 12.35	7.85 - 12.35
Anthracene	0.04 J	0.03 J
Chrysene	0.02 J	0.01 J
Pyrene	0.02 J	0.03 J

Location ID:	MW-05A	MW-05A
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	3.55 - 13.05	3.55 - 13.05
Naphthalene	0.4	< 0.07

Location ID:	MW-12A	MW-12A
Sample Date:	7/20/2018	4/15/2019
Screen Interval (ftBGS):	7.55 - 17.05	7.55 - 17.05
Anthracene	0.1	0.02 J
Benzo(a)anthracene	0.02 J	< 0.05
Chrysene	0.02 J	< 0.05
Fluoranthene	0.1	< 0.05 J
Naphthalene	0.2	0.3
Pyrene	0.09	0.02 J

Location ID:	MW-11A	MW-11A
Sample Date:	7/31/2018	4/10/2019
Screen Interval (ftBGS):	9.25 - 18.75	9.25 - 18.75
Anthracene	0.02 J	< 0.05
Chrysene	0.01 J	< 0.05

Location ID:	MW-11A DUP
Sample Date:	7/31/2018
Screen Interval (ftBGS):	9.25 - 18.75
Anthracene	0.02 J

Location ID:	MW-13A	MW-13A
Sample Date:	7/20/2018	4/14/2019
Screen Interval (ftBGS):	5.45 - 14.95	5.45 - 14.95
Pyrene	0.03 J	0.01 J

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER SVOCs (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
1-Methylnaphthalene	--	1.1	--	--	--
Anthracene	--	180	--	--	0.012
Benzo(a)anthracene	--	0.03	--	--	0.018
Chrysene	--	25	--	--	0.0018
Fluoranthene	--	80	--	--	0.04
Naphthalene	--	0.17	--	4.6	1.1
Pyrene	--	12	--	--	0.025

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

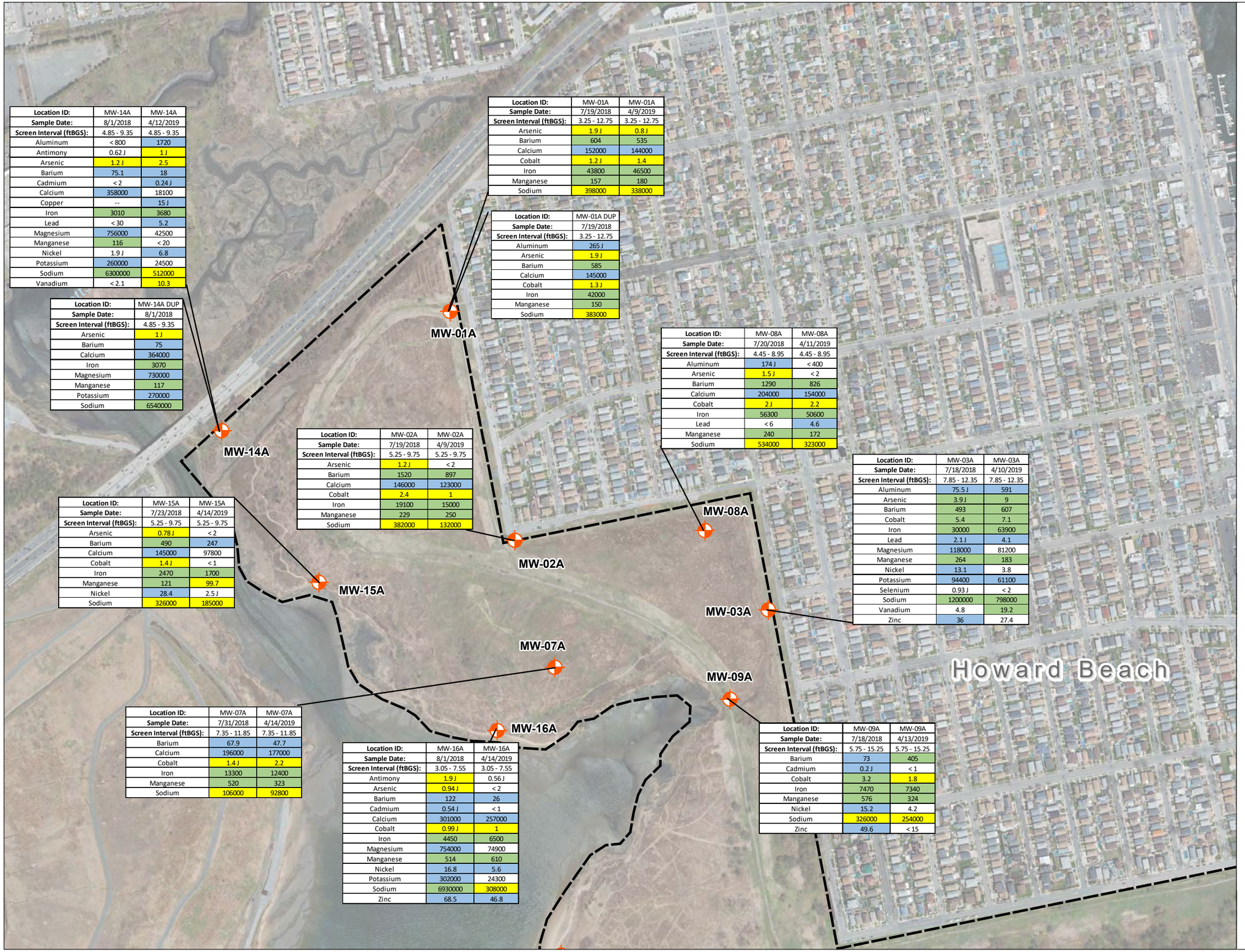
0 250 500 750 1,000
Feet

Figure 3.12: Exceedances of SVOCs in Groundwater
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.12 - Exceedances of SVOCs in Groundwater.mxd



Location ID:	MW-14A	MW-14A
Sample Date:	8/1/2018	4/12/2019
Screen Interval (ftBGS):	4.85 - 9.35	4.85 - 9.35
Aluminum	< 800	1720
Antimony	0.62 J	1 J
Arsenic	1.2 J	2.5
Barium	75.1	18
Cadmium	< 2	0.24 J
Calcium	358000	18100
Copper	--	15 J
Iron	3010	3680
Lead	< 30	5.2
Magnesium	756000	42500
Manganese	116	< 20
Nickel	1.9 J	6.8
Potassium	260000	24500
Sodium	6300000	512000
Vanadium	< 2.1	10.3

Location ID:	MW-14A DUP
Sample Date:	8/1/2018
Screen Interval (ftBGS):	4.85 - 9.35
Arsenic	1 J
Barium	75
Calcium	364000
Iron	3070
Magnesium	730000
Manganese	117
Potassium	270000
Sodium	6540000

Location ID:	MW-01A	MW-01A
Sample Date:	7/19/2018	4/9/2019
Screen Interval (ftBGS):	3.25 - 12.75	3.25 - 12.75
Arsenic	1.9 J	0.8 J
Barium	604	535
Calcium	152000	144000
Cobalt	1.2 J	1.4
Iron	43800	46500
Manganese	157	180
Sodium	398000	338000

Location ID:	MW-01A DUP
Sample Date:	7/19/2018
Screen Interval (ftBGS):	3.25 - 12.75
Aluminum	265 J
Arsenic	1.9 J
Barium	585
Calcium	145000
Cobalt	1.3 J
Iron	42000
Manganese	150
Sodium	383000

Location ID:	MW-08A	MW-08A
Sample Date:	7/20/2018	4/11/2019
Screen Interval (ftBGS):	4.45 - 8.95	4.45 - 8.95
Aluminum	174 J	< 400
Arsenic	1.5 J	< 2
Barium	1290	826
Calcium	204000	154000
Cobalt	2 J	2.2
Iron	56300	50600
Lead	< 6	4.6
Manganese	240	172
Sodium	534000	323000

Location ID:	MW-02A	MW-02A
Sample Date:	7/19/2018	4/9/2019
Screen Interval (ftBGS):	5.25 - 9.75	5.25 - 9.75
Arsenic	1.2 J	< 2
Barium	1520	897
Calcium	146000	123000
Cobalt	2.4	1
Iron	19100	15000
Manganese	229	250
Sodium	382000	132000

Location ID:	MW-03A	MW-03A
Sample Date:	7/18/2018	4/10/2019
Screen Interval (ftBGS):	7.85 - 12.35	7.85 - 12.35
Aluminum	75.5 J	591
Arsenic	3.9 J	9
Barium	493	607
Cobalt	5.4	7.1
Iron	30000	63900
Lead	2.1 J	4.1
Magnesium	118000	81200
Manganese	264	183
Nickel	13.1	3.8
Potassium	94400	61100
Selenium	0.93 J	< 2
Sodium	1200000	798000
Vanadium	4.8	19.2
Zinc	36	27.4

Location ID:	MW-15A	MW-15A
Sample Date:	7/23/2018	4/14/2019
Screen Interval (ftBGS):	5.25 - 9.75	5.25 - 9.75
Arsenic	0.78 J	< 2
Barium	490	247
Calcium	145000	97800
Cobalt	1.4 J	< 1
Iron	2470	1700
Manganese	121	99.7
Nickel	28.4	2.5 J
Sodium	326000	185000

Location ID:	MW-07A	MW-07A
Sample Date:	7/31/2018	4/14/2019
Screen Interval (ftBGS):	7.35 - 11.85	7.35 - 11.85
Barium	67.9	47.7
Calcium	196000	177000
Cobalt	1.4 J	2.2
Iron	13300	12400
Manganese	520	323
Sodium	106000	92800

Location ID:	MW-16A	MW-16A
Sample Date:	8/1/2018	4/14/2019
Screen Interval (ftBGS):	3.05 - 7.55	3.05 - 7.55
Antimony	1.9 J	0.56 J
Arsenic	0.94 J	< 2
Barium	122	26
Cadmium	0.54 J	< 1
Calcium	301000	257000
Cobalt	0.99 J	1
Iron	4450	6500
Magnesium	754000	74900
Manganese	514	610
Nickel	16.8	5.6
Potassium	302000	24300
Sodium	6930000	308000
Zinc	68.5	46.8

Location ID:	MW-09A	MW-09A
Sample Date:	7/18/2018	4/13/2019
Screen Interval (ftBGS):	5.75 - 15.25	5.75 - 15.25
Barium	73	405
Cadmium	0.2 J	< 1
Cobalt	3.2	1.8
Iron	7470	7340
Manganese	576	324
Nickel	15.2	4.2
Sodium	326000	254000
Zinc	49.6	< 15

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER METALS (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
Aluminum	--	2000	--	--	5
Antimony	6	0.78	3	--	30
Arsenic	10	0.052	25	--	3.1
Barium	2000	380	1000	--	3.9
Cadmium	5	0.92	5	--	0.07
Calcium	--	--	--	--	116000
Chromium	100	--	50	--	--
Cobalt	--	0.6	--	--	3
Copper					
Iron	--	1400	300	--	158
Lead	15	15	25	--	0.92
Magnesium	--	--	--	--	82000
Manganese	--	43	300	--	112
Nickel	--	39	100	--	5
Potassium	--	--	--	--	53000
Selenium	50	10	10	--	1
Sodium	--	--	20000	--	680000
Vanadium	--	8.6	--	--	19
Zinc	--	600	--	--	30

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

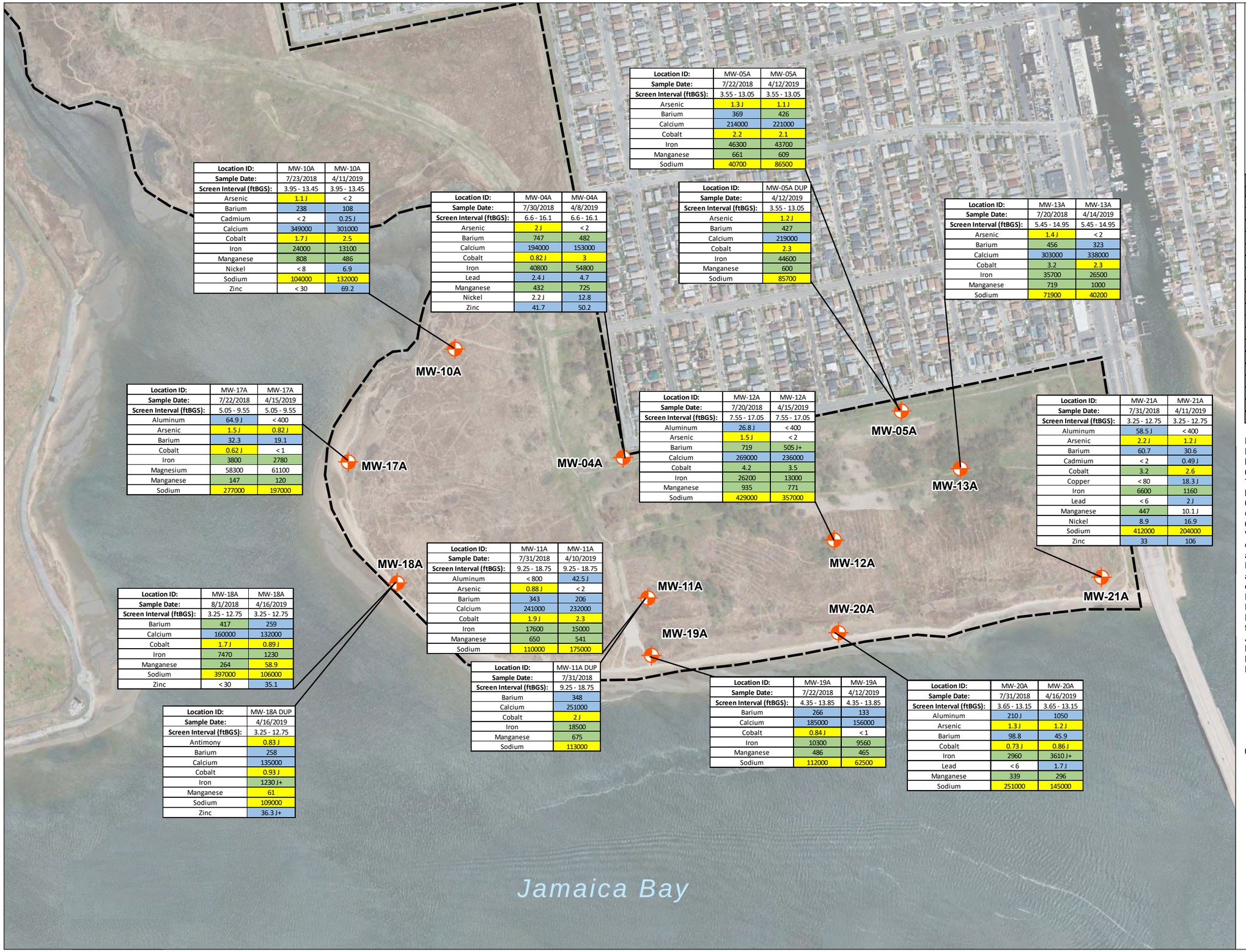
Figure 3.13a-1: Exceedances of Metals in Shallow Groundwater (North)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.13a-1 - Exceedances of Metals in Shallow Groundwater (North).mxd



Location ID:	MW-10A	MW-10A
Sample Date:	7/23/2018	4/11/2019
Screen Interval (ftBGS):	3.95 - 13.45	3.95 - 13.45
Arsenic	1.1 J	< 2
Barium	238	108
Cadmium	< 2	0.25 J
Calcium	349000	301000
Cobalt	1.7 J	2.5
Iron	24000	13100
Manganese	808	486
Nickel	< 8	6.9
Sodium	104000	132000
Zinc	< 30	69.2

Location ID:	MW-04A	MW-04A
Sample Date:	7/30/2018	4/8/2019
Screen Interval (ftBGS):	6.6 - 16.1	6.6 - 16.1
Arsenic	2 J	< 2
Barium	747	482
Calcium	194000	153000
Cobalt	0.82 J	3
Iron	40800	54800
Lead	2.4 J	4.7
Manganese	432	725
Nickel	2.2 J	12.8
Zinc	41.7	50.2

Location ID:	MW-05A	MW-05A
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	3.55 - 13.05	3.55 - 13.05
Arsenic	1.3 J	1.1 J
Barium	369	426
Calcium	214000	221000
Cobalt	2.2	2.1
Iron	46300	43700
Manganese	661	609
Sodium	40700	86500

Location ID:	MW-05A DUP
Sample Date:	4/12/2019
Screen Interval (ftBGS):	3.55 - 13.05
Arsenic	1.2 J
Barium	427
Calcium	219000
Cobalt	2.3
Iron	44600
Manganese	600
Sodium	85700

Location ID:	MW-13A	MW-13A
Sample Date:	7/20/2018	4/14/2019
Screen Interval (ftBGS):	5.45 - 14.95	5.45 - 14.95
Arsenic	1.4 J	< 2
Barium	456	323
Calcium	303000	338000
Cobalt	3.2	2.3
Iron	35700	26500
Manganese	719	1000
Sodium	71900	40200

Location ID:	MW-17A	MW-17A
Sample Date:	7/22/2018	4/15/2019
Screen Interval (ftBGS):	5.05 - 9.55	5.05 - 9.55
Aluminum	64.9 J	< 400
Arsenic	1.5 J	0.82 J
Barium	32.3	19.1
Cobalt	0.62 J	< 1
Iron	3800	2780
Magnesium	58300	61100
Manganese	147	120
Sodium	277000	197000

Location ID:	MW-12A	MW-12A
Sample Date:	7/20/2018	4/15/2019
Screen Interval (ftBGS):	7.55 - 17.05	7.55 - 17.05
Aluminum	26.8 J	< 400
Arsenic	1.5 J	< 2
Barium	719	505 J+
Calcium	269000	236000
Cobalt	4.2	3.5
Iron	26200	13000
Manganese	935	771
Sodium	429000	357000

Location ID:	MW-21A	MW-21A
Sample Date:	7/31/2018	4/11/2019
Screen Interval (ftBGS):	3.25 - 12.75	3.25 - 12.75
Aluminum	58.5 J	< 400
Arsenic	2.2 J	1.2 J
Barium	60.7	30.6
Cadmium	< 2	0.49 J
Cobalt	3.2	2.6
Copper	< 80	18.3 J
Iron	6600	1160
Lead	< 6	2 J
Manganese	447	10.1 J
Nickel	8.9	16.9
Sodium	412000	204000
Zinc	33	106

Location ID:	MW-18A	MW-18A
Sample Date:	8/1/2018	4/16/2019
Screen Interval (ftBGS):	3.25 - 12.75	3.25 - 12.75
Barium	417	259
Calcium	160000	132000
Cobalt	1.7 J	0.89 J
Iron	7470	1230
Manganese	264	58.9
Sodium	397000	106000
Zinc	< 30	35.1

Location ID:	MW-18A DUP
Sample Date:	4/16/2019
Screen Interval (ftBGS):	3.25 - 12.75
Antimony	0.83 J
Barium	258
Calcium	135000
Cobalt	0.93 J
Iron	1230 J+
Manganese	61
Sodium	109000
Zinc	36.3 J+

Location ID:	MW-11A	MW-11A
Sample Date:	7/31/2018	4/10/2019
Screen Interval (ftBGS):	9.25 - 18.75	9.25 - 18.75
Aluminum	< 800	42.5 J
Arsenic	0.88 J	< 2
Barium	343	206
Calcium	241000	232000
Cobalt	1.9 J	2.3
Iron	17600	15000
Manganese	650	541
Sodium	110000	175000

Location ID:	MW-11A DUP
Sample Date:	7/31/2018
Screen Interval (ftBGS):	9.25 - 18.75
Barium	348
Calcium	251000
Cobalt	2 J
Iron	18500
Manganese	675
Sodium	113000

Location ID:	MW-19A	MW-19A
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	4.35 - 13.85	4.35 - 13.85
Barium	266	133
Calcium	185000	156000
Cobalt	0.84 J	< 1
Iron	10300	9560
Manganese	486	465
Sodium	112000	62500

Location ID:	MW-20A	MW-20A
Sample Date:	7/31/2018	4/16/2019
Screen Interval (ftBGS):	3.65 - 13.15	3.65 - 13.15
Aluminum	210 J	1050
Arsenic	1.3 J	1.2 J
Barium	98.8	45.9
Cobalt	0.73 J	0.86 J
Iron	2960	3610 J+
Lead	< 6	1.7 J
Manganese	339	296
Sodium	251000	145000

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

Detection exceeds lowest HHSL
Detection exceeds ESL
Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER METALS (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
Aluminum	--	2000	--	--	5
Antimony	6	0.78	3	--	30
Arsenic	10	0.052	25	--	3.1
Barium	2000	380	1000	--	3.9
Cadmium	5	0.92	5	--	0.07
Calcium	--	--	--	--	116000
Chromium	100	--	50	--	--
Cobalt	--	0.6	--	--	3
Copper					
Iron	--	1400	300	--	158
Lead	15	15	25	--	0.92
Magnesium	--	--	--	--	82000
Manganese	--	43	300	--	112
Nickel	--	39	100	--	5
Potassium	--	--	--	--	53000
Selenium	50	10	10	--	1
Sodium	--	--	20000	--	680000
Vanadium	--	8.6	--	--	19
Zinc	--	600	--	--	30

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

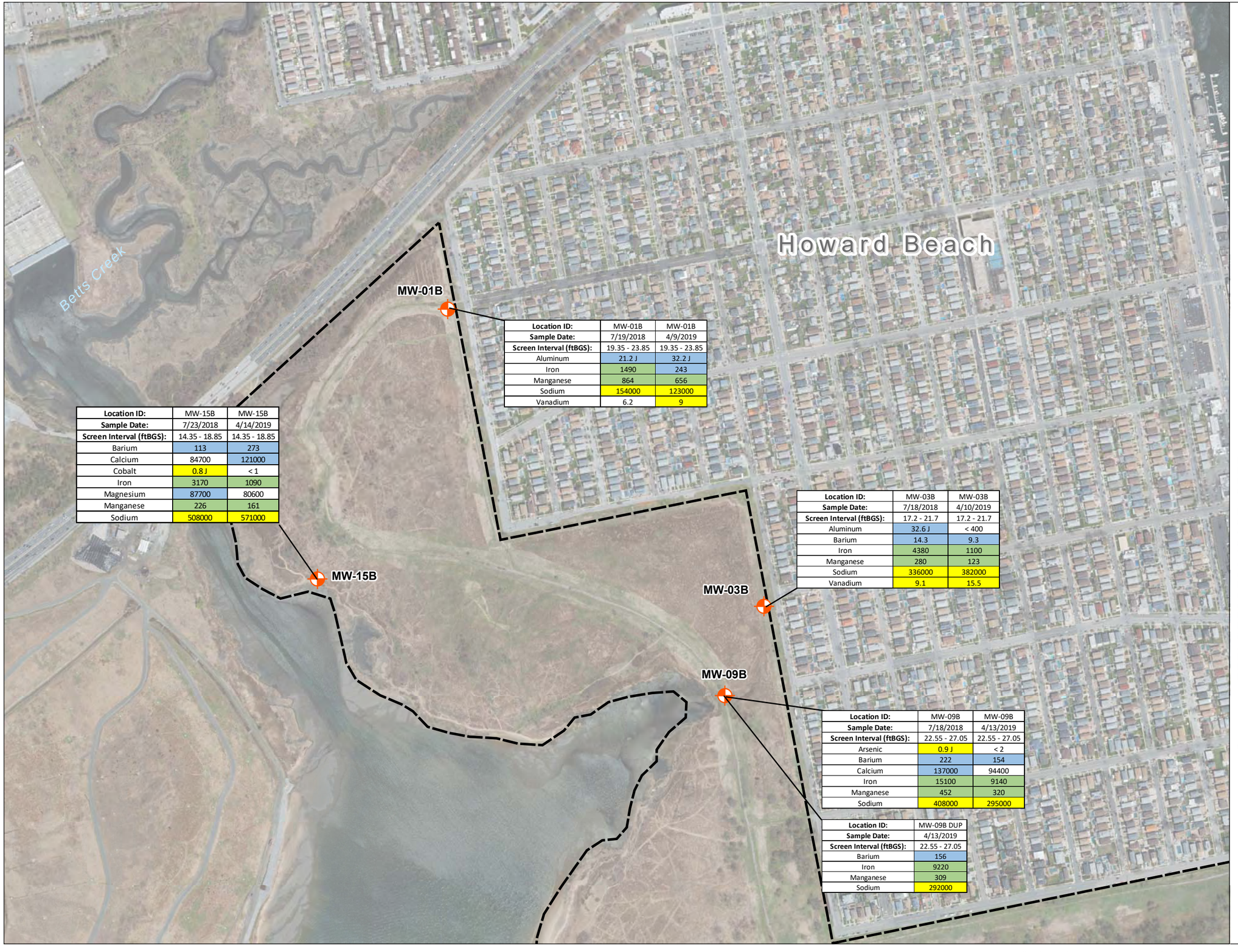
0 250 500 750 1,000
Feet

Figure 3.13a-2: Exceedances of Metals in Shallow Groundwater (South)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.13a-2 - Exceedances of Metals in Shallow Groundwater (South).mxd



Location ID:	MW-01B	MW-01B
Sample Date:	7/19/2018	4/9/2019
Screen Interval (ftBGS):	19.35 - 23.85	19.35 - 23.85
Aluminum	21.2 J	32.2 J
Iron	1490	243
Manganese	864	656
Sodium	154000	123000
Vanadium	6.2	9

Location ID:	MW-15B	MW-15B
Sample Date:	7/23/2018	4/14/2019
Screen Interval (ftBGS):	14.35 - 18.85	14.35 - 18.85
Barium	113	273
Calcium	84700	121000
Cobalt	0.8 J	< 1
Iron	3170	1090
Magnesium	87700	80600
Manganese	226	161
Sodium	508000	571000

Location ID:	MW-03B	MW-03B
Sample Date:	7/18/2018	4/10/2019
Screen Interval (ftBGS):	17.2 - 21.7	17.2 - 21.7
Aluminum	32.6 J	< 400
Barium	14.3	9.3
Iron	4380	1100
Manganese	280	123
Sodium	336000	382000
Vanadium	9.1	15.5

Location ID:	MW-09B	MW-09B
Sample Date:	7/18/2018	4/13/2019
Screen Interval (ftBGS):	22.55 - 27.05	22.55 - 27.05
Arsenic	0.9 J	< 2
Barium	222	154
Calcium	137000	94400
Iron	15100	9140
Manganese	452	320
Sodium	408000	295000

Location ID:	MW-09B DUP
Sample Date:	4/13/2019
Screen Interval (ftBGS):	22.55 - 27.05
Barium	156
Iron	9220
Manganese	309
Sodium	292000

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER METALS (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
Aluminum	--	2000	--	--	5
Arsenic	10	0.052	25	--	3.1
Barium	2000	380	1000	--	3.9
Calcium	--	--	--	--	116000
Chromium	100	--	50	--	--
Cobalt	--	0.6	--	--	3
Iron	--	1400	300	--	158
Magnesium	--	--	--	--	82000
Manganese	--	43	300	--	112
Nickel	--	39	100	--	5
Potassium	--	--	--	--	53000
Sodium	--	--	20000	--	680000
Vanadium	--	8.6	--	--	19

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.13b-1: Exceedances of Metals in Deep Groundwater (North)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.13b-1 - Exceedances of Metals in Deep Groundwater (North).mxd

Location ID:	MW-17B	MW-17B
Sample Date:	7/22/2018	4/15/2019
Screen Interval (ftBGS):	16.15 - 20.65	16.15 - 20.65
Aluminum	129 J	< 400
Barium	78.2	90.6
Calcium	131000	263000
Cobalt	3.1	1.2
Iron	17400	3610
Magnesium	202000	178000
Manganese	391	418
Potassium	86300	71000
Sodium	1630000	1410000

Location ID:	MW-19B	MW-19B
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	20.25 - 24.75	20.25 - 24.75
Aluminum	22.4 J	46.6 J
Arsenic	1.2 J	< 2
Barium	37.6	26.1
Cobalt	1.3 J	1.6
Magnesium	206000	226000
Manganese	155	268
Potassium	112000	128000
Sodium	2780000	2890000

Location ID:	MW-05B	MW-05B
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	22.85 - 27.35	22.85 - 27.35
Aluminum	< 800	41.6
Barium	58.9	17
Cobalt	1.6 J	1.9
Iron	6490	1010
Magnesium	244000	233000
Manganese	182	157
Potassium	120000	117000
Sodium	2040000	1970000
Vanadium	14.8	15.7

Location ID:	MW-06B	MW-06B
Sample Date:	7/22/2018	4/8/2019
Screen Interval (ftBGS):	15.25 - 19.75	15.25 - 19.75
Aluminum	51.9 J	34.3 J
Arsenic	0.81 J	< 2
Barium	< 8.3	5.3 J+
Cobalt	0.72 J	0.79 J
Iron	4080	4390
Manganese	260	259
Sodium	180000	178000

Location ID:	MW-21B	MW-21B
Sample Date:	7/31/2018	4/13/2019
Screen Interval (ftBGS):	19.25 - 23.75	19.25 - 23.75
Arsenic	1.1 J	< 2
Barium	31.5	19
Calcium	119000	82700
Iron	6480	8590
Manganese	116	138
Sodium	516000	490000

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER METALS (µg/L)

Analyte	MCL	TW	GA	VISL	ESV
Aluminum	--	2000	--	--	5
Arsenic	10	0.052	25	--	3.1
Barium	2000	380	1000	--	3.9
Calcium	--	--	--	--	116000
Chromium	100	--	50	--	--
Cobalt	--	0.6	--	--	3
Iron	--	1400	300	--	158
Magnesium	--	--	--	--	82000
Manganese	--	43	300	--	112
Nickel	--	39	100	--	5
Potassium	--	--	--	--	53000
Sodium	--	--	20000	--	680000
Vanadium	--	8.6	--	--	19

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI
Location data provided by
Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.13b-2: Exceedances of Metals in Deep Groundwater (South)
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.13b-2 - Exceedances of Metals in Deep Groundwater (South).mxd

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER PESTICIDES, HERBICIDES, PCBs, AND DIOXINS AND FURANS

Analyte	Units	MCL	TW	GA	VISL	ESV
beta-BHC	µg/L	--	0.025	0.04	--	2.2
Endosulfan sulfate	µg/L	--	--	--	--	0.003
Heptchlor epoxide	µg/L	0.2	0.0014	0.03	--	0.004
Dioxins and Furans TEQ	pg/L	--	0.12	0.7	--	2

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated; J-: biased low.
TEQ: Toxic Equivalent.
Dioxins and Furans concentrations via TEQ.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.14a: Exceedances of Pesticides, Herbicides, PCBs, and Dioxins and Furans in Shallow Groundwater
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.14a - Exceedances of Pesticides, Herbicides, PCBs, and Dioxins and Furans in Shallow Groundwater.mxd

Location ID:	MW-09B	MW-09B
Sample Date:	7/18/2018	4/13/2019
Screen Interval (ftBGS):	22.55 - 27.05	22.55 - 27.05
4,4-DDT (µg/L)	< 0.017	0.01 J
Total PCBs (µg/L)	< 0.0096	0.029 J

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR GROUNDWATER PESTICIDES AND PCBs

Analyte		MCL	TW	GA	VISL	ESV
4,4-DDT	µg/L	--	0.23	0.2	--	0.001
Total PCBs	µg/L	0.5	--	0.09	--	0.014

Notes:
MCL: Federal maximum contaminant level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
VISL: Vapor intrusion screening level target groundwater concentration at target risk of 1E-06 and hazard quotient 1, average groundwater temperature 25°C.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
ftBGS: feet below ground surface.
J: estimated. J-: biased low.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

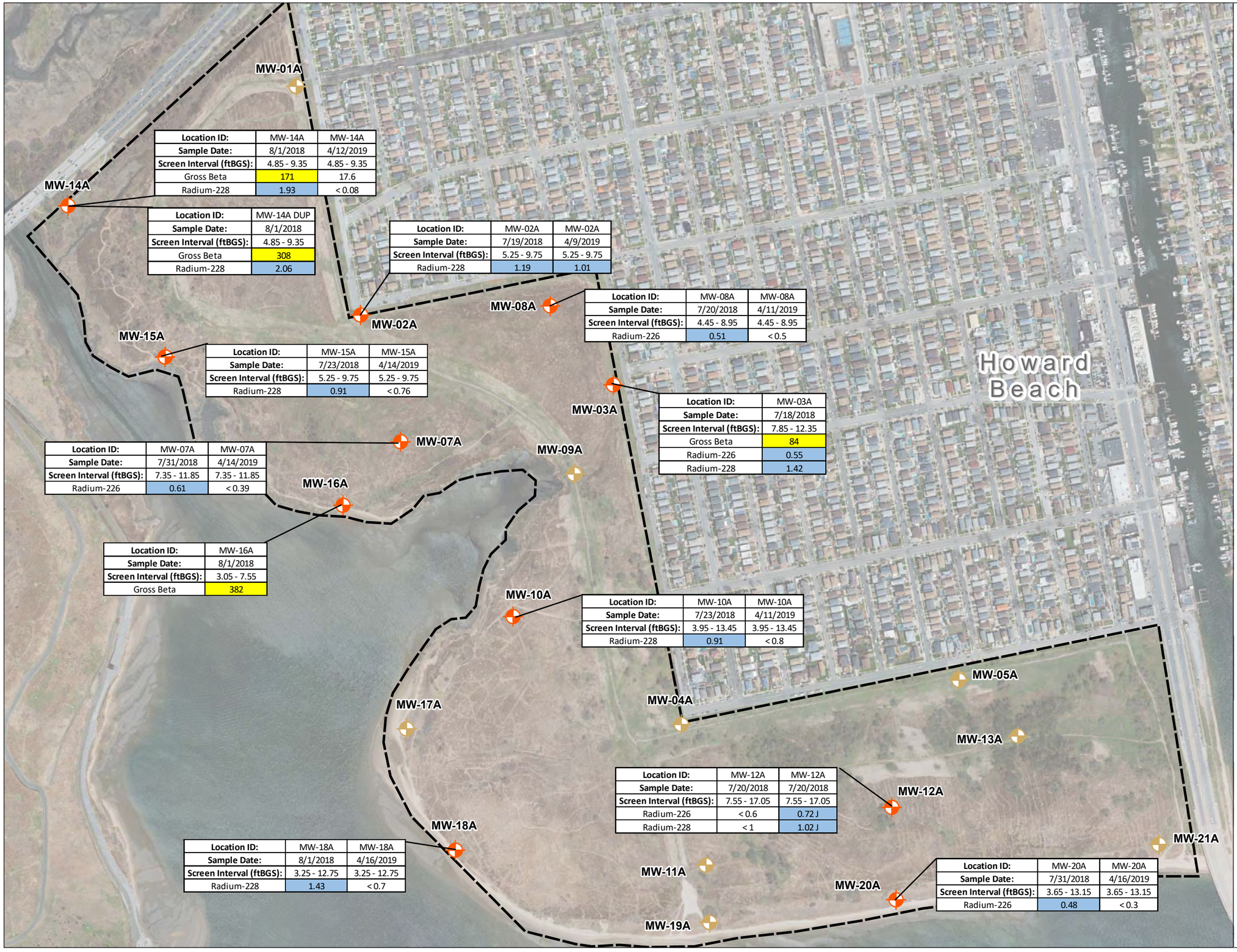
0 250 500 750 1,000
Feet

Figure 3.14b: Exceedances of Pesticides and PCBs in Deep Groundwater
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.14b - Exceedances of Pesticides, Herbicides, PCBs, and Dioxins and Furans in Deep Groundwater.mxd



Location ID:	MW-14A	MW-14A
Sample Date:	8/1/2018	4/12/2019
Screen Interval (ftBGS):	4.85 - 9.35	4.85 - 9.35
Gross Beta	171	17.6
Radium-228	1.93	< 0.08

Location ID:	MW-14A DUP
Sample Date:	8/1/2018
Screen Interval (ftBGS):	4.85 - 9.35
Gross Beta	308
Radium-228	2.06

Location ID:	MW-02A	MW-02A
Sample Date:	7/19/2018	4/9/2019
Screen Interval (ftBGS):	5.25 - 9.75	5.25 - 9.75
Radium-228	1.19	1.01

Location ID:	MW-08A	MW-08A
Sample Date:	7/20/2018	4/11/2019
Screen Interval (ftBGS):	4.45 - 8.95	4.45 - 8.95
Radium-226	0.51	< 0.5

Location ID:	MW-15A	MW-15A
Sample Date:	7/23/2018	4/14/2019
Screen Interval (ftBGS):	5.25 - 9.75	5.25 - 9.75
Radium-228	0.91	< 0.76

Location ID:	MW-03A
Sample Date:	7/18/2018
Screen Interval (ftBGS):	7.85 - 12.35
Gross Beta	84
Radium-226	0.55
Radium-228	1.42

Location ID:	MW-07A	MW-07A
Sample Date:	7/31/2018	4/14/2019
Screen Interval (ftBGS):	7.35 - 11.85	7.35 - 11.85
Radium-226	0.61	< 0.39

Location ID:	MW-16A
Sample Date:	8/1/2018
Screen Interval (ftBGS):	3.05 - 7.55
Gross Beta	382

Location ID:	MW-10A	MW-10A
Sample Date:	7/23/2018	4/11/2019
Screen Interval (ftBGS):	3.95 - 13.45	3.95 - 13.45
Radium-228	0.91	< 0.8

Location ID:	MW-12A	MW-12A
Sample Date:	7/20/2018	7/20/2018
Screen Interval (ftBGS):	7.55 - 17.05	7.55 - 17.05
Radium-226	< 0.6	0.72 J
Radium-228	< 1	1.02 J

Location ID:	MW-18A	MW-18A
Sample Date:	8/1/2018	4/16/2019
Screen Interval (ftBGS):	3.25 - 12.75	3.25 - 12.75
Radium-228	1.43	< 0.7

Location ID:	MW-20A	MW-20A
Sample Date:	7/31/2018	4/16/2019
Screen Interval (ftBGS):	3.65 - 13.15	3.65 - 13.15
Radium-226	0.48	< 0.3

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in pCi/L.

	Detection exceeds HHSL
	Detection exceeds lowest ESL
	Detection exceeds both HHSL and lowest ESL

SCREENING LEVELS FOR RADIOCHEMISTRY IN GROUNDWATER (pCi/L)

Analyte	Radionuclide Rule	Terrestrial ESV	Aquatic ESV
Radium-226	5	8110	0.1
Radium-228	5	6780	0.09
Gross Beta	50	--	--

Notes:
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

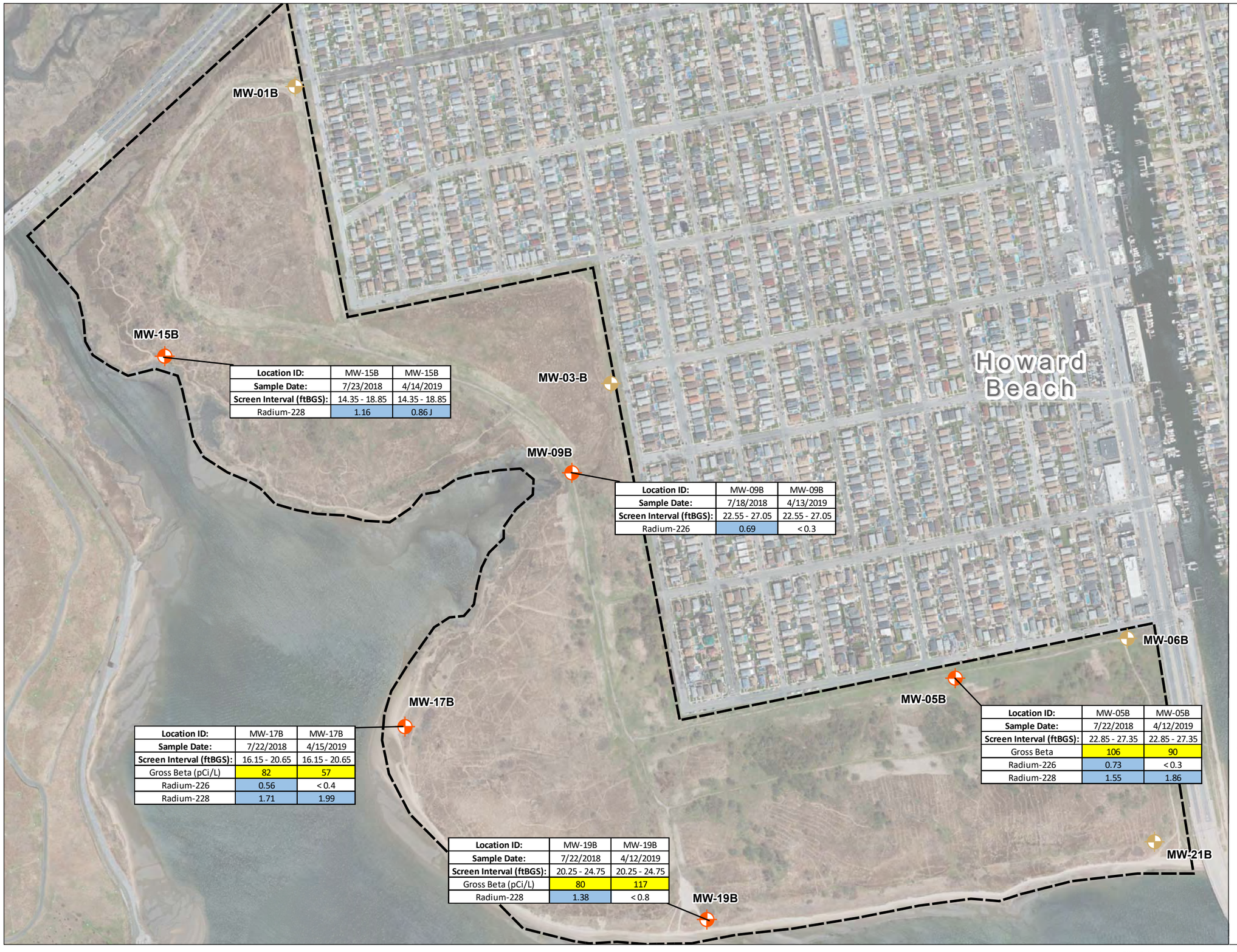
Figure 3.15a: Exceedances of Radionuclides in Shallow Groundwater
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.15a - Exceedances of Radionuclides in Shallow Groundwater.mxd



Location ID:	MW-15B	MW-15B
Sample Date:	7/23/2018	4/14/2019
Screen Interval (ftBGS):	14.35 - 18.85	14.35 - 18.85
Radium-228	1.16	0.86 J

Location ID:	MW-09B	MW-09B
Sample Date:	7/18/2018	4/13/2019
Screen Interval (ftBGS):	22.55 - 27.05	22.55 - 27.05
Radium-226	0.69	< 0.3

Location ID:	MW-17B	MW-17B
Sample Date:	7/22/2018	4/15/2019
Screen Interval (ftBGS):	16.15 - 20.65	16.15 - 20.65
Gross Beta (pCi/L)	82	57
Radium-226	0.56	< 0.4
Radium-228	1.71	1.99

Location ID:	MW-05B	MW-05B
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	22.85 - 27.35	22.85 - 27.35
Gross Beta	106	90
Radium-226	0.73	< 0.3
Radium-228	1.55	1.86

Location ID:	MW-19B	MW-19B
Sample Date:	7/22/2018	4/12/2019
Screen Interval (ftBGS):	20.25 - 24.75	20.25 - 24.75
Gross Beta (pCi/L)	80	117
Radium-228	1.38	< 0.8

Legend

- Groundwater Sample - At Least One Screening Level Exceedance
- Groundwater Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in pCi/L.

	Detection exceeds HHSL
	Detection exceeds lowest ESL
	Detection exceeds both HHSL and lowest ESL

SCREENING LEVELS FOR RADIOCHEMISTRY IN GROUNDWATER (pCi/L)

Analyte	Radionuclide Rule	Terrestrial ESV	Aquatic ESV
Radium-226	5	8110	0.1
Radium-228	5	6780	0.09
Gross Beta	50	--	--

Notes:
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
ftBGS: feet below ground surface.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.15b: Exceedances of Radionuclides in Deep Groundwater
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company

100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020

Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.15b - Exceedances of Radionuclides Deep Groundwater.mxd

Legend

- Surface Water Sample - At Least One Screening Level Exceedance
- Surface Water Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR SURFACE WATER VOCs (µg/L)

Analyte	TW	GA	ESV
Toluene	110	--	2

Notes:
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 240 480 720 960
Feet

Figure 3.16: Exceedances of VOCs in Surface Water
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.16 - Exceedances of VOCs in Surface Water.mxd

Location ID:	SW-04	SW-04
Sample Date:	7/23/2018	4/15/2019
Benzo(k)fluoranthene	0.01 J	< 0.05
Chrysene	0.02 J	< 0.05

Location ID:	SW-06	SW-06
Sample Date:	7/23/2018	4/15/2019
Chrysene	0.01 J	< 0.05

Location ID:	SW-03	SW-03
Sample Date:	7/23/2018	7/23/2018
Benzo(a)anthracene	0.02 J	< 0.05
Benzo(a)pyrene	0.02 J	< 0.05
Benzo(k)fluoranthene	0.02 J	< 0.05
Chrysene	0.04 J	0.02 J
Fluoranthene	0.05 J	0.03 J
Pyrene	0.04 J	0.02 J

Location ID:	SW-03 DUP
Sample Date:	7/23/2018
Benzo(a)anthracene	0.02 J
Benzo(a)pyrene	0.02 J
Benzo(k)fluoranthene	0.02 J
Chrysene	0.04 J
Fluoranthene	0.05 J
Pyrene	0.04 J

Location ID:	SW-07	SW-07
Sample Date:	7/23/2018	4/13/2019
Chrysene	< 0.05	0.01 J

Location ID:	SW-08	SW-08
Sample Date:	7/23/2018	4/13/2019
Chrysene	0.01 J	< 0.05

Legend

- Surface Water Sample - At Least One Screening Level Exceedance
- Surface Water Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR SURFACE WATER SVOCs (µg/L)

Analyte	TW	GA	ESV
Benzo(a)anthracene	0.03	--	0.018
Benzo(a)pyrene	0.025	--	0.014
Benzo(k)fluoranthene	2.5	--	0.0041
Chrysene	25	--	0.0018
Fluoranthene	80	--	0.04
Pyrene	12	--	0.025

Notes:
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

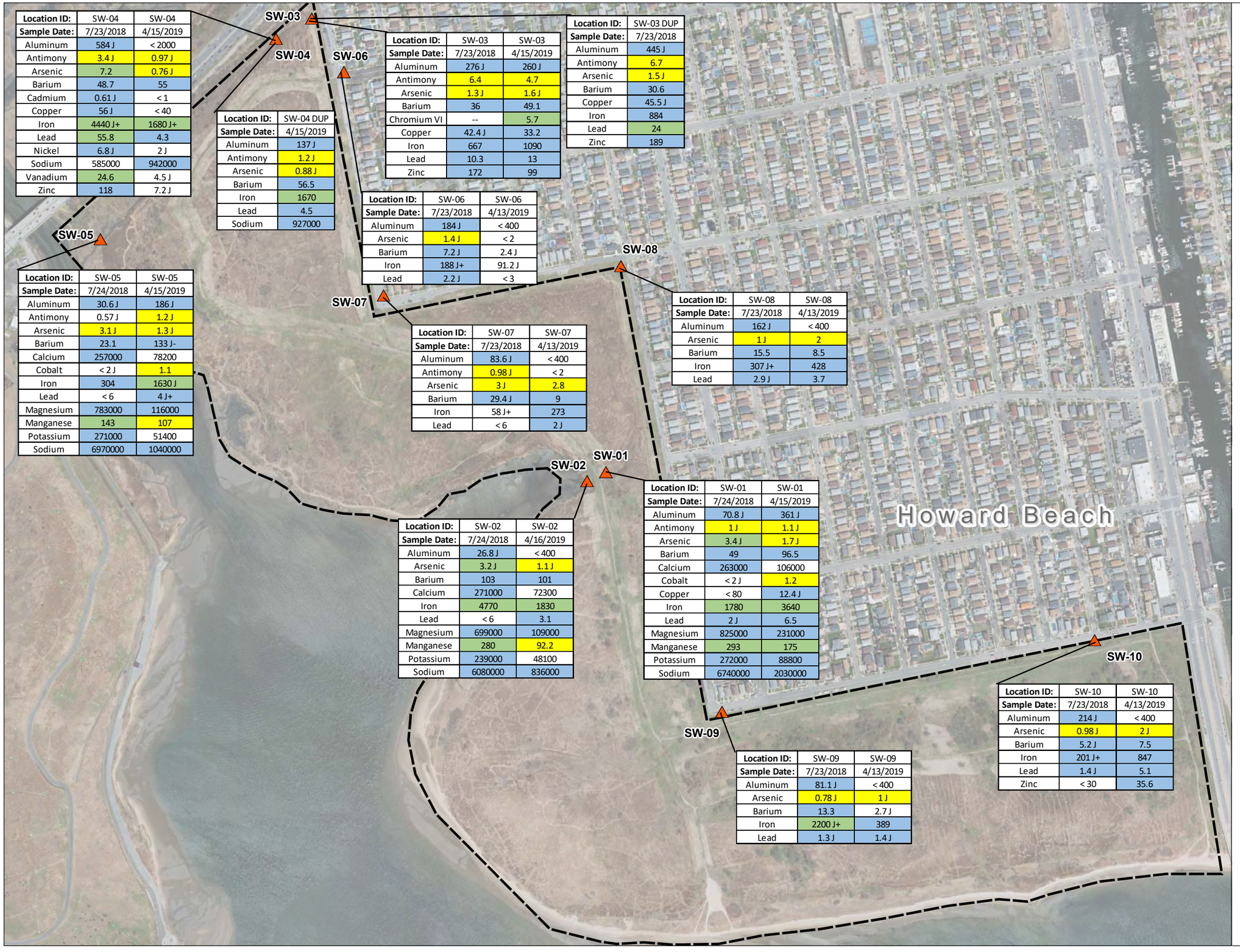
0 250 500 750 1,000
Feet

Figure 3.17: Exceedances of SVOCs in Surface Water
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.17 - Exceedances of SVOCs Surface Water.mxd



Location ID:	SW-04	SW-04
Sample Date:	7/23/2018	4/15/2019
Aluminum	584 J	< 2000
Antimony	3.4 J	0.97 J
Arsenic	7.2	0.76 J
Barium	48.7	55
Cadmium	0.61 J	< 1
Copper	56 J	< 40
Iron	4440 J+	1680 J+
Lead	55.8	4.3
Nickel	6.8 J	2 J
Sodium	585000	942000
Vanadium	24.6	4.5 J
Zinc	118	7.2 J

Location ID:	SW-04 DUP
Sample Date:	4/15/2019
Aluminum	137 J
Antimony	1.2 J
Arsenic	0.88 J
Barium	56.5
Iron	1670
Lead	4.5
Sodium	927000

Location ID:	SW-03	SW-03
Sample Date:	7/23/2018	4/15/2019
Aluminum	276 J	260 J
Antimony	6.4	4.7
Arsenic	1.3 J	1.6 J
Barium	36	49.1
Chromium VI	--	5.7
Copper	42.4 J	33.2
Iron	667	1090
Lead	10.3	13
Zinc	172	99

Location ID:	SW-03 DUP
Sample Date:	7/23/2018
Aluminum	445 J
Antimony	6.7
Arsenic	1.5 J
Barium	30.6
Copper	45.5 J
Iron	884
Lead	24
Zinc	189

Location ID:	SW-06	SW-06
Sample Date:	7/23/2018	4/13/2019
Aluminum	184 J	< 400
Arsenic	1.4 J	< 2
Barium	7.2 J	2.4 J
Iron	188 J+	91.2 J
Lead	2.2 J	< 3

Location ID:	SW-05	SW-05
Sample Date:	7/24/2018	4/15/2019
Aluminum	30.6 J	186 J
Antimony	0.57 J	1.2 J
Arsenic	3.1 J	1.3 J
Barium	23.1	133 J-
Calcium	257000	78200
Cobalt	< 2 J	1.1
Iron	304	1630 J
Lead	< 6	4 J+
Magnesium	783000	116000
Manganese	143	107
Potassium	271000	51400
Sodium	6970000	1040000

Location ID:	SW-07	SW-07
Sample Date:	7/23/2018	4/13/2019
Aluminum	83.6 J	< 400
Antimony	0.98 J	< 2
Arsenic	3 J	2.8
Barium	29.4 J	9
Iron	58 J+	273
Lead	< 6	2 J

Location ID:	SW-08	SW-08
Sample Date:	7/23/2018	4/13/2019
Aluminum	162 J	< 400
Arsenic	1 J	2
Barium	15.5	8.5
Iron	307 J+	428
Lead	2.9 J	3.7

Location ID:	SW-02	SW-02
Sample Date:	7/24/2018	4/16/2019
Aluminum	26.8 J	< 400
Arsenic	3.2 J	1.1 J
Barium	103	101
Calcium	271000	72300
Iron	4770	1830
Lead	< 6	3.1
Magnesium	699000	109000
Manganese	280	92.2
Potassium	239000	48100
Sodium	6080000	836000

Location ID:	SW-01	SW-01
Sample Date:	7/24/2018	4/15/2019
Aluminum	70.8 J	361 J
Antimony	1 J	1.1 J
Arsenic	3.4 J	1.7 J
Barium	49	96.5
Calcium	263000	106000
Cobalt	< 2 J	1.2
Copper	< 80	12.4 J
Iron	1780	3640
Lead	2 J	6.5
Magnesium	825000	231000
Manganese	293	175
Potassium	272000	88800
Sodium	6740000	2030000

Location ID:	SW-09	SW-09
Sample Date:	7/23/2018	4/13/2019
Aluminum	81.1 J	< 400
Arsenic	0.78 J	1 J
Barium	13.3	2.7 J
Iron	2200 J+	389
Lead	1.3 J	1.4 J

Location ID:	SW-10	SW-10
Sample Date:	7/23/2018	4/13/2019
Aluminum	214 J	< 400
Arsenic	0.98 J	2 J
Barium	5.2 J	7.5
Iron	201 J+	847
Lead	1.4 J	5.1
Zinc	< 30	35.6

Legend

- Surface Water Sample - At Least One Screening Level Exceedance
- Surface Water Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR SURFACE WATER TOTAL METALS (µg/L)

Analyte	TW	ESV
Aluminum	2000	5
Antimony	0.78	30
Arsenic	0.052	3.1
Barium	380	3.9
Calcium	--	116000
Chromium VI	0.04	1
Cobalt	0.6	3
Iron	1400	158
Lead	15	0.92
Magnesium	--	82000
Manganese	43	112
Nickel	39	5
Potassium	--	53000
Selenium	10	1
Sodium	--	680000
Vanadium	8.6	19
Zinc	600	30

Notes:
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
J: estimated; J+: biased high.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 250 500 750 1,000
Feet

Figure 3.18: Exceedances of Total Metals in Surface Water
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.18 - Exceedances of Total Metals in Surface Water.mxd

Location ID:	SW-04	SW-04
Sample Date:	7/23/2018	4/15/2019
Aluminum	136 J	37.3 J
Antimony	2.9 J	0.96 J
Arsenic	4.4	< 2
Barium	37.3	54.4
Iron	2850	670
Lead	7	1.1 J
Nickel	5.3 J	1.8 J
Sodium	608000	918000
Vanadium	17.5	3.3

Location ID:	SW-04 DUP
Sample Date:	4/15/2019
Aluminum	39.7 J
Antimony	0.9 J
Arsenic	0.72 J
Barium	55.9
Iron	665
Sodium	947000

Location ID:	SW-03	SW-03
Sample Date:	7/23/2018	4/15/2019
Aluminum	32.5 J	33.5 J
Antimony	5	3.6
Arsenic	0.86 J	1 J
Barium	25.2	44.4
Copper	18.7 J	18.5 J
Iron	92.6 J	258
Lead	1.5 J	2 J
Zinc	96.5	44

Location ID:	SW-03 DUP
Sample Date:	7/23/2018
Aluminum	43.2 J
Antimony	5
Arsenic	0.76 J
Barium	27.9
Copper	20.6 J
Lead	1.4 J
Zinc	98.6

Location ID:	SW-06	SW-06
Sample Date:	7/23/2018	4/13/2019
Aluminum	47.2 J	< 400
Arsenic	1.1 J	< 2
Barium	6 J	1.6 J

Location ID:	SW-05	SW-05
Sample Date:	7/24/2018	4/15/2019
Arsenic	2.9 J	< 100 J
Barium	22.1	< 200 J
Calcium	259000	262000
Iron	223	< 5000 J
Magnesium	819000	799000
Manganese	156	70.6
Potassium	290000	293000
Sodium	6820000	6720000

Location ID:	SW-07	SW-07
Sample Date:	7/23/2018	4/13/2019
Aluminum	53.5 J	< 400
Antimony	1.1 J	< 2
Arsenic	3 J	2.9
Barium	34.7 J	8.3

Location ID:	SW-08	SW-08
Sample Date:	7/23/2018	4/13/2019
Arsenic	0.81 J	1.9 J
Barium	11.3	7.8
Iron	47 J	243
Lead	< 6	1.1 J

Location ID:	SW-02	SW-02
Sample Date:	7/24/2018	4/16/2019
Arsenic	2.6 J	0.85 J
Barium	97.6	98
Calcium	267000	71300
Iron	2160	727
Magnesium	525000	107000
Manganese	255	86.1
Potassium	248000	47500
Sodium	5620000	823000

Location ID:	SW-01	SW-01
Sample Date:	7/24/2018	4/15/2019
Antimony	0.72 J	0.8 J
Arsenic	3.1 J	1.1 J
Barium	49.6	93.7
Calcium	256000	85700
Cobalt	0.25 J-	0.87 J
Iron	707	167
Magnesium	779000	177000
Manganese	269	136
Potassium	282000	67600
Sodium	6440000	1480000

Location ID:	SW-10	SW-10
Sample Date:	7/23/2018	4/13/2019
Aluminum	26.3 J	< 400
Arsenic	0.93 J	1.7 J
Barium	5.8 J	5.4
Iron	84.1 J	793

Location ID:	SW-09	SW-09
Sample Date:	7/23/2018	4/13/2019
Aluminum	23.5 J	< 400
Barium	11.1	2.2 J
Iron	596	225

Legend

- Surface Water Sample - At Least One Screening Level Exceedance
- Surface Water Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in µg/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR SURFACE WATER DISSOLVED METALS (µg/L)

Analyte	TW	ESV
Aluminum	2000	5
Antimony	0.78	30
Arsenic	0.052	3.1
Barium	380	3.9
Calcium	--	116000
Cobalt	0.6	3
Copper	80	0.23
Iron	1400	158
Magnesium	--	82000
Manganese	43	112
Nickel	39	5
Potassium	--	53000
Selenium	10	1
Sodium	--	680000
Vanadium	8.6	19
Zinc	600	30

Notes:
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
J: estimated; J+: biased high.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

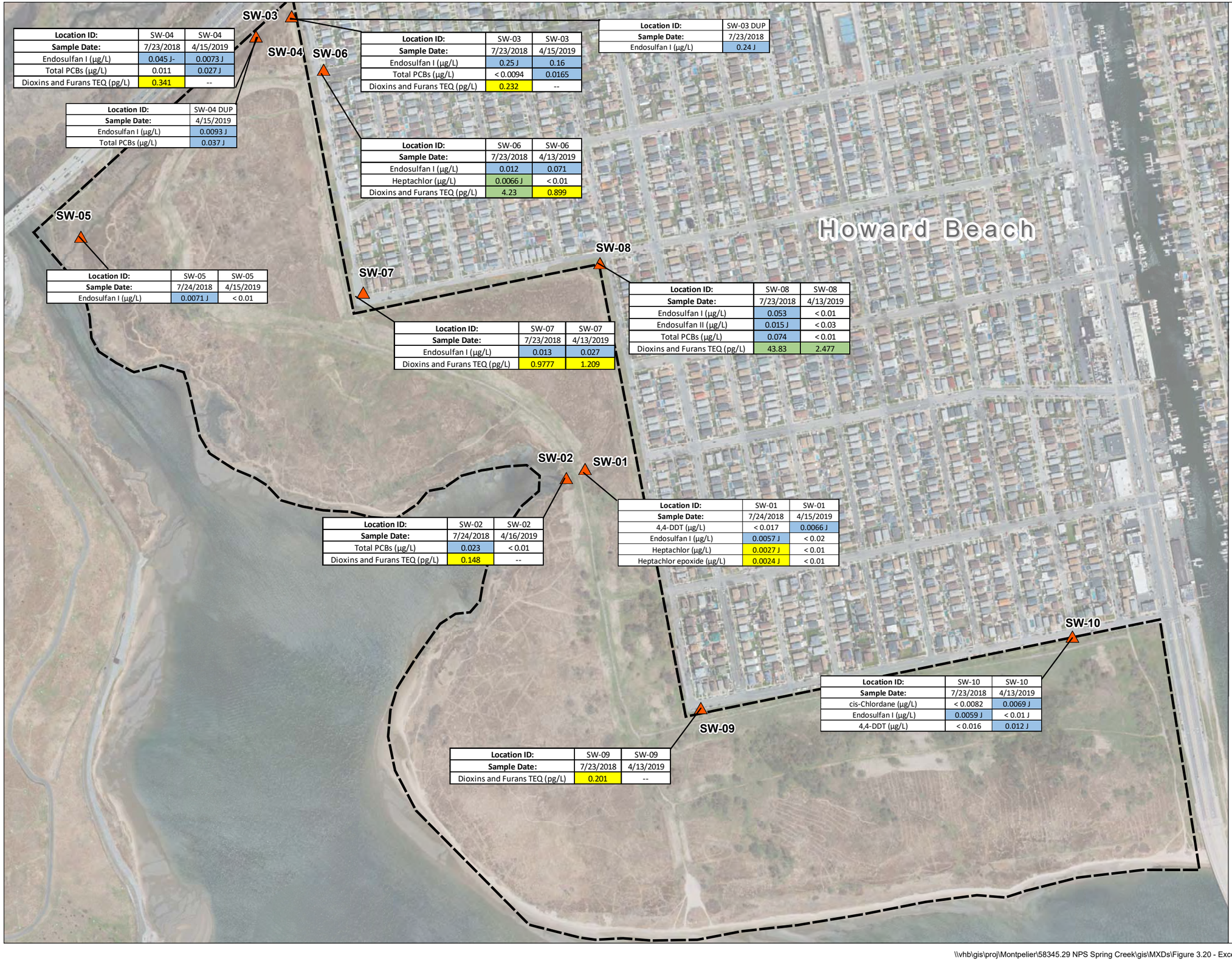
0 240 480 720 960
Feet

Figure 3.19: Exceedances of Dissolved Metals in Surface Water
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.19 - Exceedances of Dissolved Metals in Surface Water.mxd



Location ID:	SW-04	SW-04
Sample Date:	7/23/2018	4/15/2019
Endosulfan I (µg/L)	0.045 J-	0.0073 J
Total PCBs (µg/L)	0.011	0.027 J
Dioxins and Furans TEQ (pg/L)	0.341	--

Location ID:	SW-04 DUP
Sample Date:	4/15/2019
Endosulfan I (µg/L)	0.0093 J
Total PCBs (µg/L)	0.037 J

Location ID:	SW-03	SW-03
Sample Date:	7/23/2018	4/15/2019
Endosulfan I (µg/L)	0.25 J	0.16
Total PCBs (µg/L)	< 0.0094	0.0165
Dioxins and Furans TEQ (pg/L)	0.232	--

Location ID:	SW-03 DUP
Sample Date:	7/23/2018
Endosulfan I (µg/L)	0.24 J

Location ID:	SW-06	SW-06
Sample Date:	7/23/2018	4/13/2019
Endosulfan I (µg/L)	0.012	0.071
Heptachlor (µg/L)	0.0066 J	< 0.01
Dioxins and Furans TEQ (pg/L)	4.23	0.899

Location ID:	SW-05	SW-05
Sample Date:	7/24/2018	4/15/2019
Endosulfan I (µg/L)	0.0071 J	< 0.01

Location ID:	SW-08	SW-08
Sample Date:	7/23/2018	4/13/2019
Endosulfan I (µg/L)	0.053	< 0.01
Endosulfan II (µg/L)	0.015 J	< 0.03
Total PCBs (µg/L)	0.074	< 0.01
Dioxins and Furans TEQ (pg/L)	43.83	2.477

Location ID:	SW-07	SW-07
Sample Date:	7/23/2018	4/13/2019
Endosulfan I (µg/L)	0.013	0.027
Dioxins and Furans TEQ (pg/L)	0.9777	1.209

Location ID:	SW-02	SW-02
Sample Date:	7/24/2018	4/16/2019
Total PCBs (µg/L)	0.023	< 0.01
Dioxins and Furans TEQ (pg/L)	0.148	--

Location ID:	SW-01	SW-01
Sample Date:	7/24/2018	4/15/2019
4,4-DDT (µg/L)	< 0.017	0.0066 J
Endosulfan I (µg/L)	0.0057 J	< 0.02
Heptachlor (µg/L)	0.0027 J	< 0.01
Heptachlor epoxide (µg/L)	0.0024 J	< 0.01

Location ID:	SW-10	SW-10
Sample Date:	7/23/2018	4/13/2019
cis-Chlordane (µg/L)	< 0.0082	0.0069 J
Endosulfan I (µg/L)	0.0059 J	< 0.01 J
4,4-DDT (µg/L)	< 0.016	0.012 J

Location ID:	SW-09	SW-09
Sample Date:	7/23/2018	4/13/2019
Dioxins and Furans TEQ (pg/L)	0.201	--

Legend

- Surface Water Sample - At Least One Screening Level Exceedance
- Surface Water Sample - No Screening Level Exceedances
- Approximate Site Boundary

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR SURFACE WATER PESTICIDES, PCBs, AND DIOXINS AND FURANS

Analyte	Units	TW	GA	ESV
4,4-DDT	µg/L	0.23	--	0.001
cis-Chlordane	µg/L	--	--	0.0043
Endosulfan I	µg/L	--	--	0.003
Endosulfan II	µg/L	--	--	0.003
Heptachlor	µg/L	0.0014	0.04	0.0038
Heptachlor epoxide	µg/L	0.0014	0.03	0.0038
Total PCBs	µg/L	--	0.09	0.014
Dioxins and Furans TEQ	pg/L	0.12	--	2

Notes:
TW: Federal tap water screening level, regional screening level for target cancer risk of 1E-06 and hazard quotient of 0.1, June 2017 update.
GA: New York state standard for class GA water, 6 CRR-NY 703.5.
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
TEQ: Toxic Equivalent.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

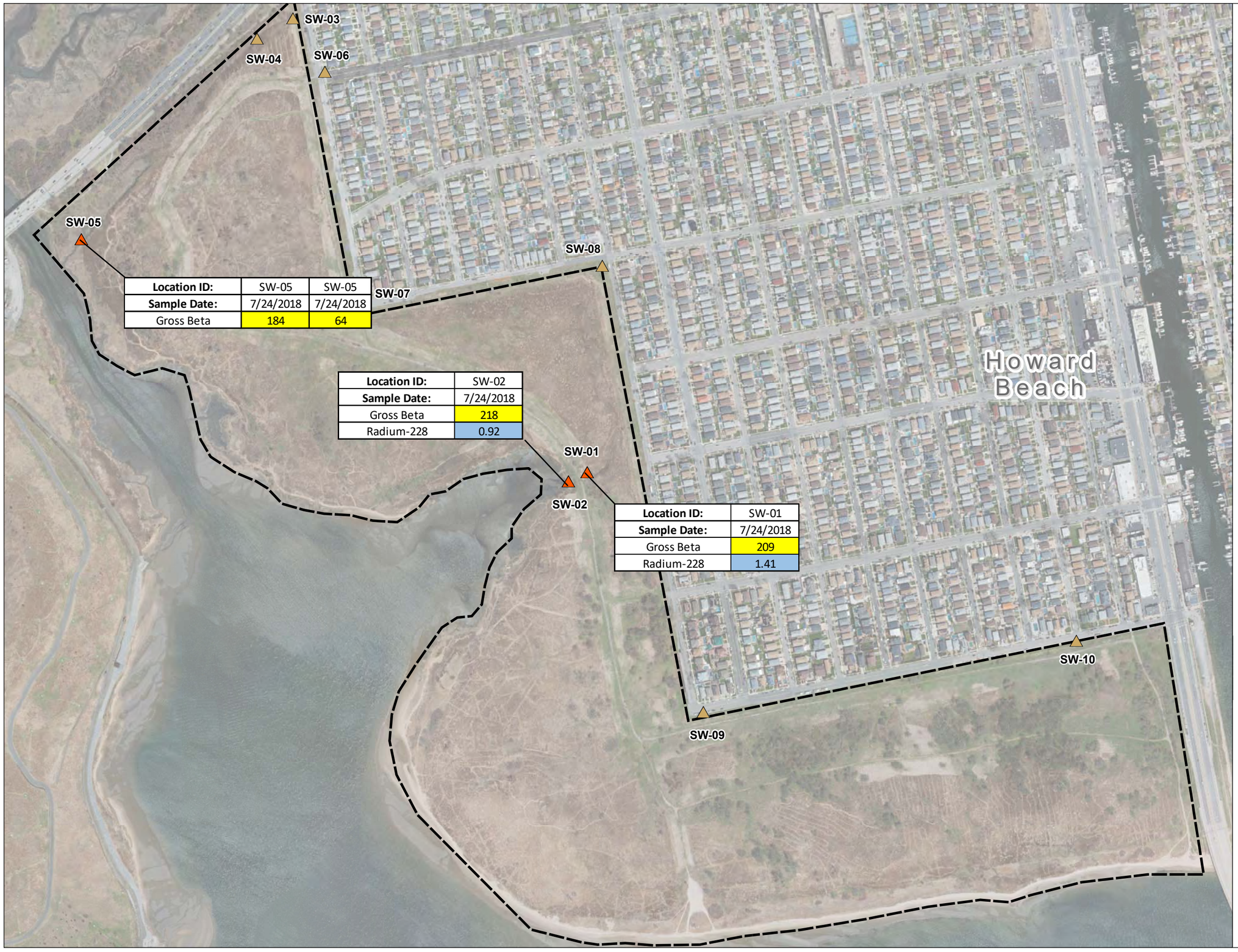
0 250 500 750 1,000
Feet

Figure 3.20: Exceedances of Pesticides, PCBs, and Dioxins and Furans in Surface Water
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.20 - Exceedances of Pesticides, PCBs, and Dioxins and Furans in Surface Water.mxd



Legend

- Surface Water Sample - At Least One Screening Level Exceedance
- Surface Water Sample - No Screening Level Exceedances
- Approximate Site Boundary

All concentrations are reported in pCi/L.

	Detection exceeds lowest HHSL
	Detection exceeds ESL
	Detection exceeds both lowest HHSL and ESL

SCREENING LEVELS FOR RADIOCHEMISTRY IN SURFACE WATER (pCi/L)

Analyte	Radionuclide Rule	Terrestrial ESV	Aquatic ESV
Radium-228	5	6780	0.09
Gross Beta	50	--	--

Notes:
ESV: 2018 NPS ecological screening value from Table 1a and Table 7 in NPS protocol for selection and use of ecological screening values for non-radiological analyses (2018).
HHSL: Human Health Screening Level.
ESL: Ecological Screening Level.
DUP: duplicate.
J: estimated.
Basemap from ESRI.
Location data provided by Rogers Surveying (August 2018).

TN

0 240 480 720 960
Feet

Figure 3.21: Exceedances of Radionuclides in Surface Water
EE/CA Investigation Summary Report
Spring Creek Park Site
Queens, New York

The Johnson Company
100 State Street, Suite 600
Montpelier, VT 05602

Drawn by: DEB Date: 09/10/2019
Reviewed by: BNH Date: 02/13/2020
Scale: 1 " = 500 ' Project: 58345.29

\\vhb\gis\proj\Montpelier\58345.29 NPS Spring Creek\gis\MXDs\Figure 3.21 - Exceedances of Radionuclides in Surface Water.mxd

Appendix B – Standard Operating Procedures

List of Standard Operating Procedures:

1. Utility Clearance
2. Logbooks
3. Records, Sample Labels, and CoCs
4. Sample Handling, Storage, and Shipping
5. IDW Management
6. Equipment Decontamination
7. Surface Water Sampling
8. Monitoring Well Installation
9. Monitoring Well Development
10. Monitoring Well Sampling
11. Soil and Rock Classification
12. Direct Push Sampling
13. Headspace Screening for VOCs
14. Operation and Calibration of a PID
15. Surface and Subsurface Soil Sampling
16. Sediment Sampling
17. Landfill Gas Screening Procedures
18. Gamma Scanning Survey
19. In Situ Gamma Spectroscopy
20. Geoprobe Dual Tube Operation

Utility Clearance

Procedure 3-01

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features at locations where planned site activities involve the physical disturbance of subsurface materials.

1.2 The procedure applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities.

1.3 The primary purpose of the procedure is to minimize the potential for damage to underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.

1.4 If there are procedures, whether it be from AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV), state, and/or federal, that are not addressed in this SOP and are applicable to utility clearance, those procedures should be added as an appendix to the project specific SAP.

1.5 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Field and subcontractor personnel shall adhere to a site-specific health and safety plan (HASP).

3.0 Terms and Definitions

3.1 **Utility**

For the proposes of this SOP, a utility is defined as a manmade underground line or conduit, cable, pipe, vault or tank that is, or was, used for the transmission of material or energy (e.g., gas, electrical, telephone, steam, water or sewage, product transfer lines, or underground storage tanks).

3.2 **As-Built Plans**

As-built plans are plans or blueprints depicting the locations of structures and associated utilities on a property.

3.3 **One-Call**

The Utility Notification Center is the one-call agency for nationwide call before you dig. The Utility Notification Center is open 24 hours a day, and accepts calls from anyone planning to dig. The phone number 811 is the designated call before you dig phone number that directly connects you to your local one-call center. Additional information can be found at www.call811.com.

Calling before you dig ensures that any publicly owned underground lines will be marked so that you can dig around them safely. Having the utility lines marked not only prevents accidental damage to the lines, but prevents property damage and personal injuries that could result in breaking a line.

The following information will need to be provided when a call is placed to One-Call:

- Your name, phone number, company name (if applicable), and mailing address.
- What type or work is being done.
- Who the work is being done for.
- The county and city the work is taking place in.
- The address or the street where the work is taking place.
- Marking instructions, (specific instructions as to where the work is taking place).

Under normal circumstances it takes between 2 to 5 days from the time you call (not counting the day of the call, weekends or holidays) to have the underground lines marked. Because these laws vary from state to state, exactly how long it will take depends on where your worksite is located. You will be given an exact start time and date when your locate request is completed, which will comply with the laws in your area.

In the event of an emergency (any situation causing damage to life or property, or a service outage), lines can be marked sooner than the original given time if requested.

3.4 **Toning**

Toning is the process of surveying an area utilizing one or more surface geophysical methods to determine the presence or absence of underground utilities. Typically, toning is conducted after identifying the general location of utilities and carefully examining all available site utility plans. Each location is marked according to the type of utility being identified. In addition, areas cleared by toning are flagged or staked to indicate that all identified utilities in a given area have been toned.

4.0 Training and Qualifications

4.1 The **Delivery Order (DO) Manager** is responsible for verifying that these utility locating procedures are performed prior to the initiation of active subsurface exploration.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all utility locating activities are performed in accordance with this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Equipment and supplies necessary for locating subsurface utilities will be provided by the subcontractor; however, the project **Field Manager/Field Personnel** will provide any additional equipment and supplies as needed as well as maintain information regarding the utility clearance activities in the field logbook.

6.0 Procedure

Proceed with the following steps where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, always exercise caution while conducting subsurface exploratory work.

6.1 **Prepare Preliminary Site Plan**

- Prepare a preliminary, scaled site plan depicting the proposed exploratory locations as part of the project specific Sampling and Analysis Plan (SAP) or Work Plan. Include as many of the cultural and natural features as practical in this plan.

6.2 **Review Background Information**

- Search existing plan files to review the as-built plans to identify the known location of utilities at the site. Plot the locations of utilities identified onto a preliminary, scaled site plan. Inform the DO Manager if utilities lie within close proximity to a proposed exploration or excavation location. The DO Manager will determine if it is necessary to relocate proposed sampling or excavation locations.
- Include the utility location information gathered during previous investigations (e.g., remedial investigation or remedial site evaluation) in the project design documents for removal or remedial actions. In this manner, information regarding utility locations collected during implementation of a DO can be shared with the subcontractor during implementation of a particular task order. In many instances, this will help to reduce the amount of additional geophysical surveying work the subcontractor may have to perform.
- Conduct interviews with onsite and facility personnel familiar with the site to obtain additional information regarding the known and suspected locations of underground utilities. In addition, if appropriate, contact shall be made with local utility companies to request their help in locating underground lines. Pencil in the dimensions, orientation, and depth of utilities, other than those identified on the as-built plans, at their approximate locations on the preliminary plans. Enter the type of utility, the personnel who provided the information, and the date the information was provided into the field log.
- During the pre-field work interviewing process, the interviewer will determine which site personnel should be notified in the event of an incident involving damage to existing utilities. Record this information in the field logbook with the corresponding telephone numbers and addresses.

6.3 Site Visit/Locate Utilities/Toning

- Prior to the initiation of field activities, the Field Task Manager or similarly qualified field personnel shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depression. Compare notes of the actual site configuration to the preliminary site plan. Note deviations in the field logbook and on the preliminary site plan. Accurately locate or survey and clearly mark with stakes, pins, flags, paint, or other suitable devices all areas where subsurface exploration is proposed. These areas shall correspond with the locations drawn on the preliminary site plan.
- Following the initial site visit by the Field Task Manager, a trained utility locating subcontractor will locate, identify, and tone all utilities depicted on the preliminary site plan. The Field Task Manager or similarly qualified field personnel shall visit the site and identify the areas of subsurface disturbance with white spray paint, chalk, white pin flags or some other easily identifiable marking. The utility locator should utilize appropriate sensing equipment to attempt to locate utilities that might not have appeared on the as-built plans. At a minimum, the utility subcontractor should utilize a metal detector and/or magnetometer; however, it is important to consider the possibility that non-metallic utilities or tanks might be present at the site. Use other appropriate surface geophysical methods such as Ground Penetrating Radar, Radiodetection, etc. as appropriate. Clear proposed exploration areas of all utilities in the immediate area where subsurface exploration is proposed. Clearly tone all anomalous areas. Clearly identify all toned areas on the preliminary site plan. All utilities near the area of subsurface disturbance should also be marked out by the utility subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). After toning the site and plotting all known or suspected buried utilities on the preliminary site plan, the utility locator shall provide the Field Task Manager with a copy of the completed preliminary site plan. Alternatively, the Field Task Manager or designee shall document the results of the survey on the preliminary site plan.
- Report to the Field Task Manager anomalous areas detected and toned that are in close proximity to the exploration or excavation areas. The Field Task Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate the proposed exploration or excavation areas. If this is required, the Field Task Manager or designee shall relocate them and clearly mark them using the methods described above. Completely remove the markings at the prior location. Plot the new locations on the site plan and delete the prior locations

from the plan. In some instances, such as in areas extremely congested with subsurface utilities, it may be necessary to dig by hand or use techniques such as air knife to determine the location of the utilities.

6.4 **Prepare Site Plan**

- Prior to the initiation of field activities, draft a final site plan that indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Provide copies of this site plan to the DO Manager and the subcontractor who is to conduct the subsurface exploration/excavation work. Review the site plan to verify its accuracy prior to initiating subsurface sampling activities.

7.0 Quality Control and Assurance

7.1 Utility locating must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 A bound field logbook will be kept detailing all activities conducted during the utility locating procedure.

8.2 The logbook will describe any changes and modifications made to the original exploration plan. The trained utility locator shall prepare a report and keep it in the project file. Also, a copy of the final site plan will be kept in the project file.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Logbooks

Procedure 3-02

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the activities and responsibilities pertaining to the identification, use, and control of logbooks and associated field data records.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 In order to keep the logbook clean, store it in a clean location and use it only when outer gloves used for PPE have been removed.

3.0 Terms and Definitions

3.1 **Logbook**

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person assigned responsibility for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 **Data Form**

A data form is a predetermined format utilized for recording field data that may become, by reference, a part of the logbook (e.g., soil boring logs, trenching logs, surface soil sampling logs, groundwater sample logs, and well construction logs are data forms).

4.0 Training and Qualifications

4.1 The **Delivery Order (DO) Manager** or **designee** is responsible for determining which team members shall record information in field logbooks and for obtaining and maintaining control of the required logbooks. The **DO Manager** shall review the field logbook on at least a monthly basis. The **DO Manager** or **designee** is responsible for reviewing logbook entries to determine compliance with this procedure and to ensure that the entries meet the project requirements.

4.2 A knowledgeable individual such as the **Field Manager**, **DO Manager**, or **Program Quality Manager** shall perform a technical review of each logbook at a frequency commensurate with the level of activity (weekly is suggested, or, at a minimum, monthly). Document these reviews by the dated signature of the reviewer on the last page or page immediately following the material reviewed.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures and that the logbook is completed properly and daily. The **Field Manager** is also responsible for submitting copies to the **DO Manager**, who is responsible for filing them and submitting a copy (if required by the DO Statement of Work).

4.5 The **logbook user** is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The **logbook user** is also responsible for safeguarding the logbook while having custody of it.

4.6 All **field personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Field logbooks shall be bound field notebooks with water-repellent pages.

5.2 Pens shall have indelible black or blue ink.

6.0 Procedure

6.1 The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Store the logbook in a clean location and use it only when outer gloves used for personal protective equipment (PPE) have been removed.

6.2 Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, include names of all samples collected in the logbook even if they are recorded elsewhere.

6.3 Enter field descriptions and observations into the logbook, as described in Attachment 1, using indelible black or blue ink.

6.4 Typical information to be entered includes the following:

- Dates (month/day/year) and times (military) of all on-site activities and entries made in logbooks/forms;
- Site name and description;
- Site location by longitude and latitude, if known;
- Weather conditions, including temperature and relative humidity;
- Fieldwork documentation, including site entry and exit times;
- Descriptions of, and rationale for, approved deviations from the work plan (WP) or field sampling plan;
- Field instrumentation readings;
- Names, job functions, and organizational affiliations of on-site personnel;
- Photograph references;
- Site sketches and diagrams made on site;
- Identification and description of sample morphology, collection locations, and sample numbers;
- Sample collection information, including dates (month/day/year) and times (military) of sample collections, sample collection methods and devices, station location numbers, sample collection depths/heights, sample preservation information, sample pH (if applicable), analysis requested (analytical groups), etc., as well as chain-of-custody (COC) information such as sample identification numbers cross-referenced to COC sample numbers;
- Sample naming convention;
- Field quality control (QC) sample information;
- Site observations, field descriptions, equipment used, and field activities accomplished to reconstruct field operations;

- Meeting information;
- Important times and dates of telephone conversations, correspondence, or deliverables;
- Field calculations;
- PPE level;
- Calibration records;
- Contractor and subcontractor information (address, names of personnel, job functions, organizational affiliations, contract number, contract name, and work assignment number);
- Equipment decontamination procedures and effectiveness;
- Laboratories receiving samples and shipping information, such as carrier, shipment time, number of sample containers shipped, and analyses requested; and
- User signatures.

6.5 The logbook shall reference data maintained in other logs, forms, etc. Correct entry errors by drawing a single line through the incorrect entry, then initialing and dating this change. Enter an explanation for the correction if the correction is more than for a mistake.

6.6 At least at the end of each day, the person making the entry shall sign or initial each entry or group of entries.

6.7 Enter logbook page numbers on each page to facilitate identification of photocopies.

6.8 If a person's initials are used for identification, or if uncommon acronyms are used, identify these on a page at the beginning of the logbook.

6.9 At least weekly and preferably daily, the **preparer** shall photocopy and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.

7.0 Quality Control and Assurance

7.1 Review per Section 4.2 shall be recorded.

8.0 Records, Data Analysis, Calculations

8.1 Retain the field logbook as a permanent project record. If a particular DO requires submittal of photocopies of logbooks, perform this as required.

8.2 Deviations from this procedure shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

9.1 Attachment 1 – Description of Logbook Entries

9.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Attachment 1
Description of Logbook Entries

Logbook entries shall be consistent with Section A.1.4 *Field Documentation SOPs* of the UFP-QAPP Manual (DoD 2005) and contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms, as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as indicated in the field sampling plan.
Weather	Indicate general weather and precipitation conditions.
Level of PPE	Record the level of PPE (e.g., Level D).
Methods	Indicate method or procedure number employed for the activity.
Sample Numbers	Indicate the unique numbers associated with the physical samples. Identify QC samples.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the WP.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
Chain of Custody and Distribution	Indicate chain-of-custody for each sample collected and indicate to whom the samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings, or photographs employed in the activity.
Narrative (including time and location)	Create a factual, chronological record of the team's activities throughout the day including the time and location of each activity. Include descriptions of general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact the work schedule, request information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself. It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

Recordkeeping, Sample Labelling, and Chain-of-Custody

Procedure 3-03

1.0 Purpose and Scope

1.1 The purpose of this standard operating procedure is to establish standard protocols for all field personnel for use in maintaining field and sampling activity records, writing sample logs, labeling samples, ensuring that proper sample custody procedures are utilized, and completing chain-of-custody/analytical request forms.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

Not applicable.

3.0 Terms and Definitions

3.1 **Logbook**

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person responsible for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 **Chain-of-Custody**

Chain-of-custody (COC) is documentation of the process of custody control. Custody control includes possession of a sample from the time of its collection in the field to its receipt by the analytical laboratory, and through analysis and storage prior to disposal.

4.0 Training and Qualifications

4.1 The **Delivery Order (DO) Manager** is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and COC forms to ensure compliance with these procedures. The **DO Manager** shall review COC forms on a monthly basis at a minimum.

4.2 The **DO Manager** and **Program Quality Manager** are responsible for evaluating project compliance with the Project Procedures Manual.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Laboratory Project Manager** or **Sample Control Department Manager** is responsible for reporting any sample documentation or COC problems to the **DO Manager** or **DO Laboratory Coordinator** within 24 hours of sample receipt.

4.5 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures. The **DO Laboratory Coordinator** is responsible for verifying that the COC/analytical request forms have been completed properly and match the sampling and analysis plan. The **DO Manager** or **DO Laboratory Coordinator** is responsible for notifying the **laboratory**, **data managers**, and **data validators** in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with

the laboratory and must be made in accordance with a respective contract (e.g., CLEAN remedial action contract).

4.6 All **field personnel** are responsible for following these procedures while conducting sampling activities. **Field personnel** are responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature.

5.0 Procedure

This procedure provides standards for documenting field activities, labeling the samples, documenting sample custody, and completing COC/analytical request forms. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

5.1 Recordkeeping

The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct each day's events. Field logs such as soil boring logs and ground-water sampling logs will also be used. These procedures are described in Procedure 3-02, *Logbooks*.

5.2 Sample Labeling

Affix a sample label with adhesive backing to each individual sample container. Place clear tape over each label (preferably prior to sampling) to prevent the labels from tearing off, falling off, being smeared, and to prevent loss of information on the label. Record the following information with a waterproof marker on each label:

- Project name or number (optional);
- COC sample number;
- Date and time of collection;
- Sampler's initials;
- Matrix (optional);
- Sample preservatives (if applicable); and
- Analysis to be performed on sample (this shall be identified by the method number or name identified in the subcontract with the laboratory).

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels.

5.3 Custody Procedures

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. Maintain custody of samples in accordance with the U.S. Environmental Protection Agency (EPA) COC guidelines prescribed in EPA *NEIC Policies and Procedures*, National Enforcement Investigations Center, Denver, Colorado, revised May 1986; EPA *RCRA Ground Water Monitoring Technical Enforcement Guidance Document* (TEGD); *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA OSWER Directive 9355 3-01); Appendix 2 of the *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*; and *Test Methods for Evaluating Solid Waste* (EPA SW-846)

A description of sample custody procedures is provided below.

5.3.1 Sample Collection Custody Procedures

According to the U.S. EPA guidelines, a sample is considered to be in custody if one of the following conditions is met:

- It is in one's actual physical possession or view;
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal);
- It is retained in a secured area with restricted access; and
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

Place custody seals on sample containers immediately after sample collection and on shipping coolers if the cooler is to be removed from the sampler's custody. Place custody seals in such a manner that they must be broken to open the containers or coolers. Label the custody seals with the following information:

- Sampler's name or initials; and
- Date and time that the sample/cooler was sealed.

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment 1.

Field personnel shall also log individual samples onto COC forms (carbon copy or computer generated) when a sample is collected. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 5.4, indicating sample identification number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The **samplers** will also sign the COC form signifying that they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the **person delivering the samples for transport** will sign and indicate the date and time on the accompanying COC form. One copy of the COC form will be retained by the **sampler** and the remaining copies of the COC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique COC form. Whenever a transfer of custody takes place, **both parties** shall sign and date the accompanying carbon copy COC forms, and the **individual relinquishing the samples** shall retain a copy of each form. One exception is when the samples are shipped; the **delivery service personnel** will not sign or receive a copy because they do not open the coolers. The **laboratory** shall attach copies of the completed COC forms to the reports containing the results of the analytical tests. An example COC form is provided in Attachment 2.

5.3.2 Laboratory Custody Procedures

The following custody procedures are to be followed by an **independent laboratory** receiving samples for chemical analysis; the procedures in their Naval Facilities Engineering Service Center-evaluated Laboratory Quality Assurance Plan must follow these same procedures. A **designated sample custodian** shall take custody of all samples upon their arrival at the analytical laboratory. The **custodian** shall inspect all sample labels and COC forms to ensure that the information is consistent, and that each is properly completed. The **custodian** will also measure the temperature of the temperature blank in the coolers upon arrival using either a National Institute for Standards and Technology calibrated thermometer or an infra-red temperature gun. The **custodian** shall note the condition of the samples including:

- If the samples show signs of damage or tampering;
- If the containers are broken or leaking;
- If headspace is present in sample vials;
- If proper preservation of samples has occurred (made by pH measurement, except volatile organic compounds [VOCs] and purgeable total petroleum hydrocarbons [TPH] and temperature). The pH of VOC and purgeable TPH samples will be checked by the **laboratory analyst** after the sample aliquot has been removed from the vial for analysis; and
- If any sample holding times have been exceeded.

All of the above information shall be documented on a sample receipt sheet by the **custodian**.

Discrepancies or improper preservation shall be noted by the **laboratory** as an out-of-control event and shall be documented on an out-of-control form with corrective action taken. The out-of-control form shall be signed and dated by the **sample control custodian** and **any other persons** responsible for corrective action. An example of an out-of-control form is included as Attachment 4.

The **custodian** shall then assign a unique laboratory number to each sample and distribute the samples to secured storage areas maintained at 4 degrees Celsius (soil samples for VOC analysis are to be stored in a frozen state until analysis). The unique laboratory number for each sample, COC sample number, client name, date and time received, analysis due date, and storage shall also be manually logged onto a sample receipt record and later entered into the laboratory's computerized data management system. The **custodian** shall sign the shipping bill and maintain a copy.

Laboratory personnel shall be responsible for the care and custody of samples from the time of their receipt at the laboratory through their exhaustion or disposal. Samples should be logged in and out on internal laboratory COC forms each time they are removed from storage for extraction or analysis.

5.4 Completing COC/Analytical Request Forms

COC form/analytical request form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment 2 is an example of a generic COC/analytical request form that may be used by **field personnel**. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Attachment 3 is an example of a completed site-specific COC/analytical request form, with box numbers identified and discussed in text below.

COC forms tailored to each DO can be drafted and printed onto multi-ply forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; TAT; and the same general comments each time.

Complete one COC form per cooler. Whenever possible, place all VOC analyte vials into one cooler in order to reduce the number of trip blanks. Complete all sections and be sure to sign and date the COC form. One copy of the COC form must remain with the field personnel.

Box 2 **Bill To:** List the name and address of the person/company to bill only if it is not in the subcontract with the laboratory.

Box 3 **Sample Disposal Instructions:** These instructions will be stated in the Master Service Agreement or each DO statement of work with each laboratory.

Shipment Method: State the method of shipment (e.g., hand carry or air courier via FedEx or DHL).

Comments: This area shall be used by the field team to communicate observations, potential hazards, or limitations that may have occurred in the field or additional information regarding analysis (e.g., a specific metals list, samples expected to contain high analyte concentrations).

Box 4 **Cooler No.:** This will be written on the inside or outside of the cooler and shall be included on the COC. Some laboratories attach this number to the trip blank identification, which helps track samples for VOC analysis. If a number is not on the cooler, field personnel shall assign a number, write it on the cooler, and write it on the COC.

QC Level: Enter the reporting quality control (QC) requirements (e.g., Full Data Package, Summary Data Package).

Turnaround time (TAT): TAT will be determined by a sample delivery group (SDG), which may be formed over a 14-day period, not to exceed 20 samples. Once the SDG has been completed, standard TAT is 21 calendar days from receipt of the last sample in the SDG. Entering NORMAL or STANDARD in this field will be acceptable. If quicker TAT is required, it shall be in the subcontract with the laboratory and reiterated on each COC to remind the laboratory.

Box 5 **Type of Containers:** Write the type of container used (e.g., 1-liter glass amber, for a given parameter in that column).

Preservatives: Field personnel must indicate on the COC the correct preservative used for the analysis requested. Indicate the pH of the sample (if tested) in case there are buffering conditions found in the sample matrix.

Box 6 **Sample Identification (ID) Number:** This is typically a five-character alphanumeric identifier used by the contractor to identify samples. The use of this identifier is important since the laboratories are restricted to the number of characters they are able to use. Sample numbering shall be in accordance with the project-specific sampling and analysis plan.

Description (Sample ID): This name will be determined by the location and description of the sample, as described in the project-specific sampling and analysis plan. This sample identification should not be submitted to the laboratory, but should be left blank. If a computer COC version is used, the sample identification can be input, but printed with this block black. A cross-referenced list of the COC Sample Number and sample identification must be maintained separately.

Date Collected: Record the collection date in order to track the holding time of the sample. Note: For trip blanks, record the date it was placed in company with samples.

Time Collected: When collecting samples, record the time the sample is first collected. Use of the 24-hour military clock will avoid a.m. or p.m. designations (e.g., 1815 instead of 6:15 p.m.). Record local time; the laboratory is responsible for calculating holding times to local time.

Lab ID: This is for laboratory use only.

Box 7 **Matrix/QC:** Identify the matrix (e.g., water, soil, air, tissue, fresh water sediment, marine sediment, or product). If a sample is expected to contain high analyte concentrations (e.g., a tank bottom sludge or distinct product layer), notify the laboratory in the comment section. Mark an "X" for the sample(s) that have extra volume for laboratory QC matrix spike/matrix spike duplicate (MS/MSD) purposes. The sample provided for MS/MSD purposes is usually a field duplicate.

Box 8 **Analytical Parameters:** Enter the parameter by descriptor and the method number desired (e.g., BTEX 8260B, PAHs 8270C, etc.). Whenever practicable, list the parameters as they appear in the laboratory subcontract to maintain consistency and avoid confusion.

If the COC does not have a specific box for number of sample containers, use the boxes below the analytical parameter, to indicate the number of containers collected for each parameter.

Box 9 **Sampler's Signature:** The person who collected samples must sign here.

Relinquished By: The person who turned over the custody of the samples to a second party other than an express mail carrier, such as FedEx or DHL, must sign and date here.

Received By: Typically, a representative of the receiving laboratory signs and dates here. Or, a field crew member who delivered the samples in person from the field to the laboratory might sign here. A courier, such as FedEx or DHL, does not sign here because they do not open the coolers. It must also be used by the prime contracting laboratory when samples are to be sent to a subcontractor.

Relinquished By: In the case of subcontracting, the primary laboratory will sign and date the Relinquished By space and fill out an additional COC to accompany the samples being subcontracted.

Received By (Laboratory): This space is for the final destination (e.g., at a subcontracted laboratory). A representative of the final destination (e.g., subcontracted laboratory) must sign and date here.

Box 10 **Lab No. and Questions:** This box is to be filled in by the laboratory only.

Box 11 **Control Number:** This number is the "COC" followed by the first contractor identification number in that cooler, or contained on that COC. This control number must be unique (i.e., never used twice). Record the date the COC is completed. It should be the same date the samples are collected.

Box 12 **Total # of Containers:** Sum the number of containers in that row.

Box 13 **Totals:** Sum the number of containers in each column. Because COC forms contain different formats depending on who produced the form, not all of the information listed in items 1 to 13 may be recorded; however, as much of this information as possible shall be included.

6.0 Quality Control and Assurance

6.1 Recordkeeping, sample labeling, and chain-of-custody activities must incorporate quality control measures to ensure accuracy and completeness.

6.2 Deviations from this procedure or the project-specific DO work plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

7.0 Records, Data Analysis, Calculations

7.1 The COC/analytical request form shall be faxed approximately daily to the **DO Laboratory Coordinator** for verification of accuracy. Following the completion of sampling activities, the sample logbook and COC

forms will be transmitted to the **DO Manager** for storage in project files. The **data validators** shall receive a copy also. The original COC/analytical request form shall be submitted by the **laboratory** along with the data delivered. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in the records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 Attachment 1 – Chain-of-Custody Seal

8.2 Attachment 2 – Generic Chain-of-Custody/Analytical Request Form

8.3 Attachment 3 – Sample Completed Chain-of-Custody

8.4 Attachment 4 – Sample Out-of-Control Form

8.5 Environmental Protection Agency, United States (EPA). 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA.* Interim Final. EPA/540/G-89/004. Office of Emergency and Remedial Response. October.

8.6 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

8.7 EPA. 1997. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. 3rd ed., Final Update IIIA. Office of Solid Waste.

8.8 Water Resources Control Board, State of California. 1988. *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*. August.

8.9 Procedure 3-02, *Logbooks.*

Attachment 1
Example Chain-of-Custody Seal

CHAIN-OF-CUSTODY SEAL

<table>
<tr><td rowspan="3">[LABORATORY]</td><td>SAMPLE NO.</td><td>DATE</td><td>SEAL BROKEN BY</td></tr>
<tr><td colspan="2">SIGNATURE</td><td>DATE</td></tr>
<tr><td colspan="3">PRINT NAME AND TITLE (Inspector, Analyst or Technician</td></tr>
</table>

Attachment 2
Generic Chain-of-Custody / Analytical Request Form

M901376

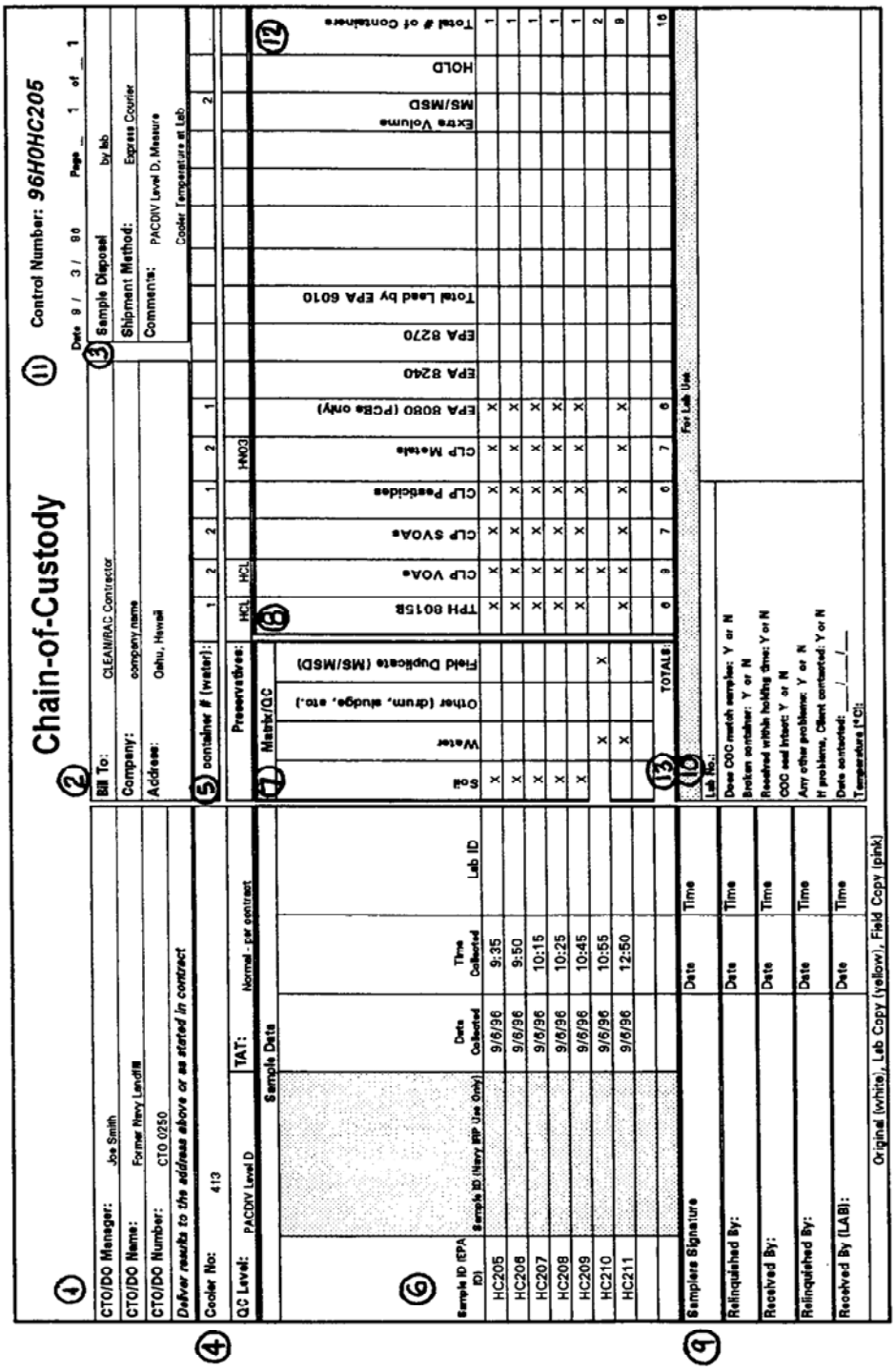
CHAIN OF CUSTODY RECORD Page ____ of ____

Client/Project Name:	Project Location:	Analysis Requested
Project Number:	Field Logbook No.:	
Sampler: (Print Name) /Affiliation:	Chain of Custody Tape No.:	
Signature:	Send Results/Report to:	

Field Sample No./ Identification	Date	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered							Lab I.D.	Remarks

Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	Analytical Laboratory (Destination):
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	Serial No.

Attachment 3
Sample Completed Chain-of-Custody



Chain-of-Custody

Control Number: 96H0HC205

CTO/DO Manager: Joe Smith
CTO/DO Name: Former Navy Landfill
CTO/DO Number: CTO 0250
Deliver results to the address above or as stated in contract
Cooler No: 413
QC Level: PACDIV Level D
TAT: Normal - per contract

Bill To: CLEAN/RAC Contractor
Company: company name
Address: Oahu, Hawaii
Sample Disposal: by lab
Shipment Method: Express Courier
Comments: PACDIV Level D, Measure Cooler Temperature at Lab

Date 9 / 3 / 96 Page 1 of 1

Sample ID (EPA ID)	Sample ID (Navy EMP Use Only)	Date Collected	Time Collected	Lab ID	Soil	Water	Other (drum, sludge, etc.)	Field Duplicate (MS/MSD)	TPH 8015B	CLP VOAs	CLP SVOAs	CLP Pesticides	CLP Metals	EPA 8080 (PCBs only)	EPA 8240	EPA 8270	Total Lead by EPA 6010	Extra Volume MS/MSD	HOLD	Total # of Containers
									HCL	HCL			HNO3							
									1	2	2	1	2	1				2		
HC205		9/6/96	9:35		X				X	X	X	X	X	X						1
HC206		9/6/96	9:50		X				X	X	X	X	X	X						1
HC207		9/6/96	10:15		X				X	X	X	X	X	X						1
HC208		9/6/96	10:25		X				X	X	X	X	X	X						1
HC209		9/6/96	10:45		X				X	X	X	X	X	X						1
HC210		9/6/96	10:55			X		X		X										2
HC211		9/6/96	12:50			X			X	X	X	X	X	X						8
									0	8	7	6	7	6						16

TOTALS

For Lab Use:
Lab No.:
Does COC match samples: Y or N
Broken container: Y or N
Received within holding time: Y or N
COC seal intact: Y or N
Any other problems: Y or N
If problems, Client contacted: Y or N
Date contacted: ___/___/___
Temperature (°C):

Samplers Signature — Date — Time
Relinquished By: — Date — Time
Received By: — Date — Time
Relinquished By: — Date — Time
Received By (LAB): — Date — Time

Original (white), Lab Copy (yellow), Field Copy (pink)

Attachment 4
Sample Out-of-Control Form

OUT OF CONTROL FORM	Status	Date	Initial
	Noted OOC		
	Submit for CA*		
	Resubmit for CA*		
	Completed		

Date Recognized:	By:
Dated Occurred:	Matrix
Parameter (Test Code):	Method:
Analyst:	Supervisor:

Samples Affected (List by Accession AND Sample No.)

1. Type of Event (Check all that apply)		2. Corrective Action (CA)* (Check all that apply)	
	Calibration Corr. Coefficient <0.995		Repeat calibration
	%RSD>20%		Made new standards
	Blank >MDL		Reran analysis
	Does not meet criteria:		Sample(s) redigested and rerun
	Spike		Sample(s) reextracted and rerun
	Duplicate		Recalculated
	LCS		Cleaned system
	Calibration Verification		Ran standard additions
	Standard Additions		Notified
	MS/MSD		Other (please explain)
	BS/BSD		
	Surrogate Recovery		
	Calculations Error		
	Holding Times Missed		
	Other (Please explain	Comments:	

3. Results of Corrective Action	
	Return to Control (indicated with)
	Corrective Actions Not Successful - DATA IS TO BE FLAGGED with _____.

Analyst:	Date:
Supervisor:	Date:
QA Department:	Date:

Sample Handling, Storage, and Shipping

Procedure 3-04

1.0 Purpose and Scope

1.1 This standard operating procedure describes the actions to be used by personnel engaged in handling, storing, and transporting samples. The objective is to obtain samples of actual conditions with as little alteration as possible.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Avoid lifting heavy coolers with back muscles; instead, use leg muscles or dollies.

2.2 Wear proper gloves, such as blue nitrile and latex, as defined in the project-specific health and safety plan, when handling sample containers to avoid contacting any materials that may have spilled out of the sample containers.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Delivery Order (DO) Manager** and the **Laboratory Project Manager** are responsible for identifying instances of non-compliance with this procedure and ensuring that future sample transport activities comply with this procedure.

4.2 The **Field Manager** is responsible for ensuring that all samples are shipped according to this procedure.

4.3 **Field personnel** are responsible for the implementation of this procedure.

4.4 The **Program Quality Manager** is responsible for ensuring that sample handling, storage, and transport activities conducted during all DOs comply with this procedure.

4.5 All **field personnel** are responsible for the implementation of this procedure.

5.0 Procedure

5.1 **Handling and Storage**

Immediately following collection, label all samples according to Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*. The lids of the containers shall not be sealed with duct tape, but may be covered with custody seals or placed directly into self-sealing bags. Place the sample containers in an insulated cooler with frozen gel packs (e.g., “blue ice”) or ice in double, sealed self-sealing bags. Samples should occupy the lower portion of the cooler, while the ice should occupy the upper portion. Place an absorbent material (e.g., proper absorbent cloth material) on the bottom of the cooler to contain liquids in case of spillage. Fill all empty space between sample containers with Styrofoam® “peanuts” or other appropriate material. Prior to shipping, wrap glass sample containers on the sides, tops, and bottoms with bubble wrap or other appropriate padding and/or surround them in Styrofoam to prevent breakage during transport. Pack all glass containers for water samples in an upright position, never stacked or on their sides. Prior to shipment, replace the ice or cold packs in the coolers so that samples will be maintained as close to 4 degrees Celsius (°C) as possible from the time of collection through transport to the analytical laboratory. Ship samples within 24 hours or on a schedule allowing the laboratory to meet holding times for analyses. The procedures for maintaining sample temperatures at 4°C pertain to all field samples.

5.2 Shipping

Follow all appropriate U.S. Department of Transportation regulations (e.g., 49 Code of Federal Regulations [CFR], Parts 171-179) for shipment of air, soil, water, and other samples. Elements of these procedures are summarized below.

5.2.1 Hazardous Materials Shipment

Field personnel must state whether any sample is suspected to be a hazardous material. A sample should be assumed hazardous unless enough evidence exists to indicate it is non-hazardous. If not suspected to be hazardous, shipments may be made as described in the Section 5.2.2 for non-hazardous materials. If hazardous, follow the procedures summarized below.

Any substance or material that is capable of posing an unreasonable risk to life, health, or property when transported is classified as hazardous. Perform hazardous materials identification by checking the list of dangerous goods for that particular mode of transportation. If not on that list, materials can be classified by checking the Hazardous Materials Table (49 CFR 172.102 including Appendix A) or by determining if the material meets the definition of any hazard class or division (49 CFR Part 173), as listed in Attachment 2.

All **persons shipping hazardous materials** must be properly trained in the appropriate regulations, as required by HM-126F, Training for Safe Transportation of Hazardous Materials (49 CFR HM-126F Subpart H). The training covers loading, unloading, handling, storing, and transporting of hazardous materials, as well as emergency preparedness in the case of accidents. **Carriers**, such as commercial couriers, must also be trained. Modes of shipment include air, highway, rail, and water.

When shipping hazardous materials, including bulk chemicals or samples suspected of being hazardous, the proper shipping papers (49 CFR 172 Subpart C), package marking (49 CFR 172 Subpart D), labeling (49 CFR 172 Subpart E), placarding (49 CFR 172 Subpart F, generally for carriers), and packaging must be used. Attachment 1 shows an example of proper package markings. Refer to a copy of 49 CFR each time hazardous materials/potentially hazardous samples are shipped.

According to Section 2.7 of the International Air Transport Association Dangerous Goods Regulations publication, very small quantities of certain dangerous goods may be transported without certain marking and documentation requirements as described in 49 CFR Part 172; however, other labeling and packing requirements must still be followed. Attachment 2 shows the volume or weight for different classes of substances. A “Dangerous Goods in Excepted Quantities” label must be completed and attached to the associated shipping cooler (Attachment 3). Certain dangerous goods are not allowed on certain airlines in any quantity.

As stated in item 4 of Attachment 4, the Hazardous Materials Regulations do not apply to hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), and sodium hydroxide (NaOH) added to water samples if their pH or percentage by weight criteria is met. These samples may be shipped as non-hazardous materials as discussed below.

5.2.2 Non-Hazardous Materials Shipment

If the samples are suspected to be non-hazardous based on previous site sample results, field screening results, or visual observations, if applicable, then samples may be shipped as non-hazardous.

When a cooler is ready for shipment to the laboratory, place two copies of the chain-of-custody form inside a self-sealing bag and tape it to the inside of the insulated cooler. Then, seal the cooler with waterproof tape and label it with “Fragile,” “This-End-Up” (or directional arrows pointing up), or other appropriate notices. Place chain-of-custody seals on the coolers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*.

5.2.3 Shipments from Outside the Continental United States

Shipment of sample coolers to the United States from locations outside the continental United States is controlled by the U.S. Department of Agriculture (USDA) and is subject to their inspection and regulation. A “USDA Soil Import Permit” is required to prove that the receiving analytical laboratory is certified by the USDA to receive and properly dispose of soil. In addition, all sample coolers must be inspected by a **USDA representative**, affixed with a label indicating that the coolers contain environmental samples, and accompanied by shipping forms stamped by the **USDA inspector** prior to shipment.

In addition, the U.S. Customs Service must clear samples shipped from U.S. territorial possessions or foreign countries upon entry into the United States. As long as the commercial invoice is properly completed (see below), shipments typically pass through U.S. Customs Service without the need to open coolers for inspection.

Completion and use of proper paperwork will, in most cases, minimize or eliminate the need for the USDA and U.S. Customs Service to inspect the contents. Attachment 5 shows an example of how paperwork may be placed on the outside of coolers for non-hazardous materials. For hazardous materials, refer to Section 5.2.1.

In summary, tape the paperwork listed below to the outside of the coolers to accompany sample shipments. If a shipment is made up of multiple pieces (e.g., more than one cooler), the paperwork need only be attached to one cooler, provided that the **courier** agrees. All other coolers in the shipment need only to be taped and have the address and chain-of-custody seals affixed.

1. **Courier Shipping Form & Commercial Invoice:** See Attachment 6 and Attachment 7 for examples of the information to be included on the commercial invoices for soil and water, respectively. Place the courier shipping form and commercial invoice inside a clear, plastic, adhesive-backed pouch that adheres to the package (typically supplied by the courier) and place it on the cooler lid as shown in Attachment 5.
2. **Soil Import Permit (soil only):** See Attachment 8 and Attachment 9 for examples of the soil import permit and soil samples restricted entry labels, respectively. The **laboratory** shall supply these documents prior to mobilization. The USDA often stops shipments of soil without these documents. Staple together the 2-inch × 2-inch USDA label (described below) and soil import permit, and place them inside a clear plastic pouch. The **courier** typically supplies the clear, plastic, adhesive-backed pouches that adhere to the package.

   Placing one restricted entry label as shown in Attachment 5 (covered with clear packing tape) and one stapled to the actual permit is suggested.

   The USDA does not control water samples, so the requirements for soil listed above do not apply.
3. **Chain-of-Custody Seals:** The **laboratory** should supply the seals. **DO personnel** must sign and date these. At least two seals should be placed in such a manner that they stick to both the cooler lid and body. Placing the seals over the tape (as shown in Attachment 5), then covering it with clear packing tape is suggested. This prevents the seal from coming loose and enables detection of tampering.
4. **Address Label:** Affix a label stating the destination (laboratory address) to each cooler.
5. **Special Requirements for Hazardous Materials:** See Section 5.2.1.

Upon receipt of sample coolers at the laboratory, the **sample custodian** shall inspect the sample containers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*. The samples shall then be immediately extracted and/or analyzed, or stored in a refrigerated storage area until they are removed for extraction and/or analysis. Whenever the samples are not being extracted or analyzed, they shall be returned to refrigerated storage.

6.0 Quality Control and Assurance

6.1 Sample handling, storage, and shipping must incorporate quality control measures to ensure conformance to these and the project requirements.

7.0 Records, Data Analysis, Calculations

7.1 Maintain records as required by implementing these procedures.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or Reference

8.1 Attachment 1 – Example Hazardous Material Package Marking

8.2 Attachment 2 – Packing Groups

8.3 Attachment 3 – Label for Dangerous Goods in Excepted Quantities

8.4 Attachment 4 – SW-846 Preservative Exception

8.5 Attachment 5 – Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States

8.6 Attachment 6 – Commercial Invoice – Soil

8.7 Attachment 7 – Commercial Invoice – Water

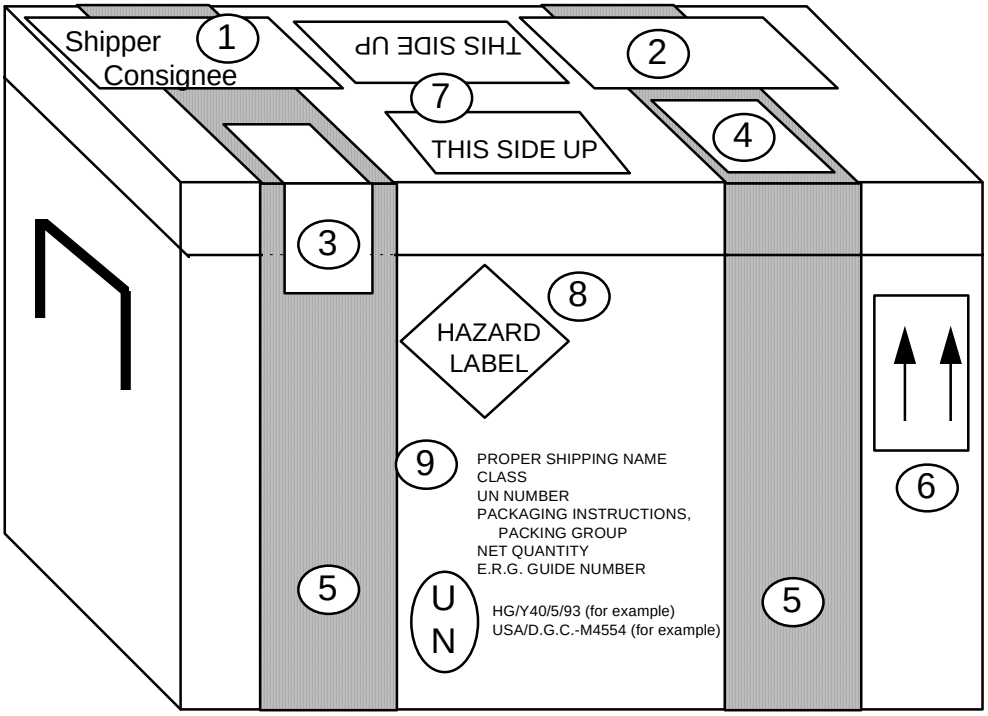
8.8 Attachment 8 – Soil Import Permit

8.9 Attachment 9 – Soil Samples Restricted Entry Labels

8.10 *DoD Environmental Field Sampling Handbook Revision 1.0*, April 2013.

8.11 Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*.

Attachment 1
Example Hazardous Material Package Marking



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**
7. **THIS SIDE UP STICKERS**
8. **HAZARD LABEL**
9. **HAZARDOUS MATERIAL INFORMATION**
10. **PACKAGE SPECIFICATIONS**

Attachment 2
Packing Groups

PACKING GROUP OF THE SUBSTANCE	PACKING GROUP 1		PACKING GROUP II		PACKING GROUP III	
CLASS or DIVISION of PRIMARY or SUBSIDIARY RISK	Packagings		Packagings		Packagings	
	Inner	Outer	Inner	Outer	Inner	Outer
1: Explosives	---------- Forbidden (Note A) ----------					
2.1: Flammable Gas	---------- Forbidden (Note B) ----------					
2.2: Non-Flammable, non-toxic gas	---------- See Notes A and B ----------					
2.3: Toxic gas	---------- Forbidden (Note A) ----------					
3. Flammable liquid	30 mL	300 mL	30 mL	500 mL	30 mL	1 L
4.1 Self-reactive substances	Forbidden		Forbidden		Forbidden	
4.1: Other flammable solids	Forbidden		30 g	500 g	30 g	1 kg
4.2: Pyrophoric substances	Forbidden		Not Applicable		Not Applicable	
4.2 Spontaneously combustible substances	Not Applicable		30 g	500 g	30 g	1 kg
4.3: Water reactive substances	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.1: Oxidizers	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.2: Organic peroxides (Note C)	See Note A		30 g or 30 mL	500 g or 250 mL	Not Applicable	
6.1: Poisons - Inhalation toxicity	Forbidden		1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - oral toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - dermal toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.2: Infectious substances	---------- Forbidden (Note A) ----------					
7: Radioactive material (Note D)	---------- Forbidden (Note A) ----------					
8: Corrosive materials	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
9: Magnetized materials	---------- Forbidden (Note A) ----------					
9: Other miscellaneous materials (Note E)	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L

Note A: *Packing groups are not used for this class or division.*
Note B: *For inner packagings, the quantity contained in receptacle with a water capacity of 30 mL. For outer packagings, the sum of the water capacities of all the inner packagings contained must not exceed 1 L.*
Note C: *Applies only to Organic Peroxides when contained in a chemical kit, first aid kit or polyester resin kit.*
Note D: *Samples will be shipped in accordance with DOT regulations for excepted packaged, as necessary.*
Note E: *For substances in Class 9 for which no packing group is indicated in the List of Dangerous Goods, Packing Group II quantities must be used.*

Attachment 3
Dangerous Goods in Excepted Quantities

Attachment 4
SW-846 Preservative Exception

Measurement	Vol. Req. (mL)	Container[2]	Preservative[3,4]	Holding Time[5]
MBAS	250	P, G	Cool, 4ºC	48 Hours
NTA	50	P, G	Cool, 4ºC	24 Hours

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.

2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

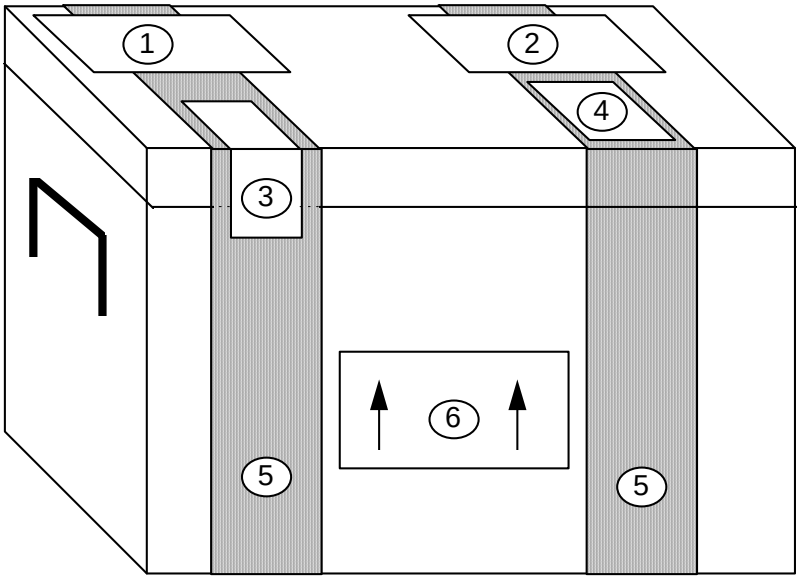
3. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4ºC until compositing and sample splitting is completed.

4. When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. for the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentration of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

6. Should only be used in the presence of residual chlorine.

Attachment 5
Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**

Attachment 6
Commercial Invoice – Soil

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <DO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	
INTERNATIONAL AIR WAYBILL NO.	(NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Soil samples for laboratory analysis only				$1.00	$3.00

TOTAL NO. OF PKGS.	TOTAL WEIGHT	TOTAL INVOICE VALUE
3		$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94
Name/Title	Signature	Date

Attachment 7
Commercial Invoice – Water

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <DO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	
INTERNATIONAL AIR WAYBILL NO.	(NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Water samples for laboratory analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

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SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94

Attachment 8
Soil Import Permit

UNITED STATES DEPARTMENT OF AGRICULTURE

Animal and Plant Health Inspection Service

Plant Protection and Quarantine

Soil Permit

Permit Number: S-52299

Issued To:

Columbia Analytical Services
(Lee Wolf)
1317 S. 13th Avenue
Kelso, Washington 98626

TELEPHONE: (360) 577-7222

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not heat treated at the port of entry, only if a compliance agreement (PPQ Form 519) has been completed and signed. Compliance Agreements and Soil permits are non-transferable. If you hold a Soil Permit and you leave your present employer or company, you must notify your local USDA office promptly.
2. To be shipped in sturdy, leakproof, containers.
3. To be released without treatment at the port of entry.
4. To be used only for analysis and only in the facility of the permittee at Columbia Analytical Services, located in Kelso, Washington.
5. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
6. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or heat treated by the permittee at the conclusion of the project as approved and prescribed by Plant Protection and Quarantine.
7. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry.

JUNE 30, 2006
Expiration Date

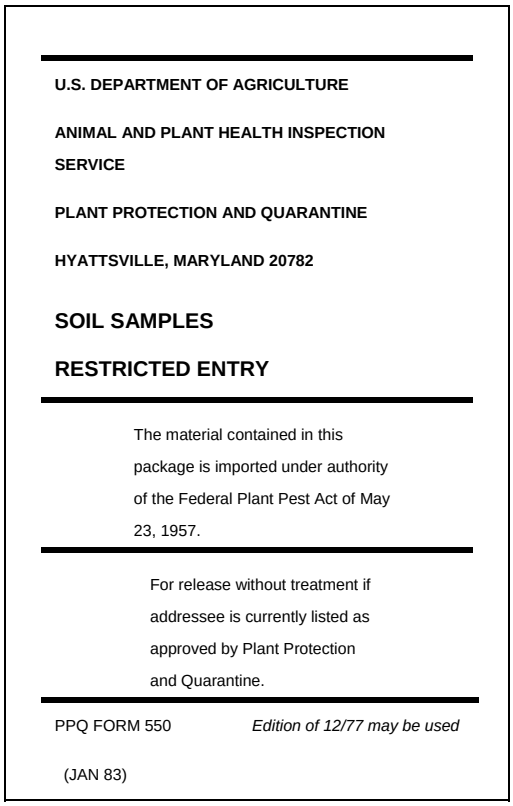
Approving Official DEBORAH M. KNOTT

WARNING: Any alteration, forgery, or unauthorized use of this Federal form is subject to civil penalties of up to $250,000 (7 U.S.C. s 7734(b)) or punishable by a fine of not more than $10,000, or imprisonment of not more than 5 years, or both (18 U.S.C. s 1001).

PPQ FORM 525B (8/94)

Pt. 1 - PERMITTEE

Attachment 9
Soil Samples Restricted Entry Labels



U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

SOIL SAMPLES

RESTRICTED ENTRY

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550 *Edition of 12/77 may be used*

(JAN 83)

Investigation Derived Waste Management

Procedure 3-05

1.0 Purpose and Scope

This standard operating procedure (SOP) describes activities and responsibilities with regard to management of investigation-derived waste (IDW). The purpose of this procedure is to provide guidance for the minimization, handling, labelling, temporary storage, inventory, classification, and disposal of IDW. This procedure will also apply to personal protective equipment (PPE), sampling equipment, decontamination fluids, non-IDW trash, non-indigenous IDW, and hazardous waste generated during implementation of removal or remedial actions. The information presented will be used to prepare and implement work plans (WPs) for IDW-related field activities. The results from implementation of WPs will then be used to develop and implement final IDW disposal plans.

If there are procedures whether it be from AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV), state and/or federal that are not addressed in this SOP and are applicable to IDW then those procedures may be added as an appendix to the project specific SAP.

This procedure shall serve as management-approved professional guidance for the LSI Sampling Plan and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Delivery Order (DO) Manager and the Quality Assurance (QA) Manager or Technical Director, and documented.

This procedure was developed to serve as management-approved professional guidance for the management of IDW generated under LSI Sampling Plan. It focuses on the requirements for minimizing, segregating, handling, labeling, storing, and inventorying IDW in the field. Certain drum inventory requirements related to the screening, sampling, classification, and disposal of IDW are also noted in this procedure.

2.0 Safety

The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the DO WP and/or direction from the **Site Safety Officer (SSO)**.

All **Field Personnel** responsible for IDW management must adhere to the HASP and must wear the PPE specified in the site-specific HASP. Generally, this includes, at a minimum, steel-toed boots or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). If safe alternatives are not achievable, discontinue site activities immediately.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **DO Manager** is responsible for ensuring that IDW management activities comply with this procedure. The **DO Manager** is responsible for ensuring that all personnel involved in IDW management shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all IDW is managed according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

The equipment and supplies required for implementation of this SOP include the following:

- Containers for waste (e.g., [U.S. Department of Transportation] DOT approved 55-gallon open and closed top drums) and material to cover waste to protect from weather (e.g., plastic covering);
- Hazardous /non-hazardous waste drum labels (weatherproof);
- Permanent marking pens;
- Inventory forms for project file;
- Plastic garbage bags, zip lock storage bags, roll of plastic sheeting; and
- Steel-toed boots, chemical resistant gloves, coveralls, safety glasses, and any other PPE required in the HASP.

6.0 Procedure

The following procedures are used to handle the IDW.

6.1 Drum Handling

6.1.1 IDW shall be containerized using DOT approved drums. The drums shall be made of steel or plastic, have a 55-gallon capacity, be completely painted or opaque, and have removable lids (i.e., United Nations Code 1A2 or 1H2). Typically 55-gallon drums are used, however small drums may be used depending on the amount of waste generated. New steel drums are preferred over recycled drums.

6.1.2 Recycled drums should not be used for hazardous waste, PCBs or other regulated shipments. For short-term storage of liquid IDW prior to discharge, double-walled bulk steel or plastic storage tanks may be used. For this scenario, consider the scheduling and cost-effectiveness of this type of bulk storage, treatment, and discharge system versus longer-term drum storage.

6.1.3 For long-term IDW storage at other project locations, the DOT approved drums with removable lids are recommended. Verify the integrity of the foam or rubber sealing ring located on the underside of some drum lids prior to sealing drums containing IDW liquids.

6.1.4 If the ring is only partially attached to the drum lid, or if a portion of the ring is missing, select another drum lid with a sealing ring that is in sound condition.

6.1.5 To prepare IDW drums for labeling, wipe clean the outer wall surfaces and drum lids of all material that might prevent legible and permanent labeling. If potentially contaminated material adheres to the outer surface of a drum, wipe that material from the drum, and segregate the paper towel or rag used to remove the material with visibly soiled PPE and disposable sampling equipment. Label all IDW drums and place them on pallets prior to storage.

6.2 Labelling

6.2.1 Containers used to store IDW must be properly labelled. Two general conditions exist: 1) from previous studies or on-site data, waste characteristics are known to be either hazardous or nonhazardous; or 2) waste characteristics are unknown until additional data are obtained.

6.2.2 For situations where the waste characteristics are known, the waste containers should be packaged and labelled in accordance with state regulations and any federal regulations that may govern the labelling of waste.

6.2.3 The following information shall be placed on all non-hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.4 The following information shall be placed on all hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Generator information (i.e., name, address, contact telephone number);
- EPA identification number (supplied by on-site client representative);
- Date when the waste was first accumulated.

6.2.5 When the final characterization of a waste is unknown, a notification label should be placed on the drum with the words "waste characterization pending analysis" and the following information included on the label:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.6 Once the waste has been characterized, the label should be changed as appropriate for a nonhazardous or hazardous waste.

6.2.7 Waste labels should be constructed of a weatherproof material and filled out with a permanent marker to prevent being washed off or becoming faded by sunlight. It is recommended that waste labels be placed on the side of the container, since the top is more subject to weathering. However, when multiple containers are accumulated together, it also may be helpful to include labels on the top of the containers to facilitate organization and disposal.

6.2.8 Each container of waste generated shall be recorded in the field notebook used by the person responsible for labelling the waste. After the waste is disposed of, either by transportation off-site or disposal on-site in an approved disposal area, an appropriate record shall be made in the same field notebook to document proper disposition of IDW.

6.3 Types of Site Investigation Waste

Several types of waste are generated during site investigations that may require special handling. These include solid, liquid, and used PPE, as discussed further below.

Solid Waste

Soil cuttings from boreholes will typically be placed in containers unless site specific requirements allow for soil cuttings to be placed back into the borehole after drilling is complete. Drilling mud generated during investigation activities shall be collected in containers. Covers should be included on the containers and must be secured at all times and only open during filling activities. The containers shall be labelled in accordance with this SOP. An inventory containing the source, volume, and description of material put in the containers shall be logged on prescribed forms and kept in the project file.

Non-hazardous solid waste can be disposed on-site in the designated site landfill or in a designated evaporation pond if it is liquefied. Hazardous wastes must be disposed off-site at an approved hazardous waste landfill.

Liquid Waste

Groundwater generated during monitoring well development, purging, and sampling can be collected in truck-mounted containers and/or other transportable containers (i.e., 55-gallon drums). Lids or bungs on drums must be secured at all times and only open during filling or pumping activities. The containers shall be labelled in accordance with this SOP. Non-hazardous liquid waste can be disposed of in one of the designated lined evaporation ponds on-site. Hazardous wastes must be handled separately and disposed off-site at an approved hazardous waste facility.

Personal Protective Equipment

PPE that is generated throughout investigation activities shall be placed in plastic garbage bags. If the solid or liquid waste that was being handled is characterized as hazardous waste, then the

corresponding PPE should also be disposed as hazardous waste. If not, all PPE should be disposed as non-hazardous waste in the designated on-site landfill. Trash that is generated as part of field activities may be disposed of in the landfill as long as the trash was not exposed to hazardous media.

6.4 Waste Accumulation On-Site

6.4.1 Solid, liquid, or PPE waste generated during investigation activities that are classified as nonhazardous or "characterization pending analysis" should be disposed of as soon as possible. Until disposal, such containers should be inventoried, stored as securely as possible, and inspected regularly, as a general good practice.

6.4.2 Solid, liquid, or PPE waste generated during investigation activities that are classified as hazardous shall not be accumulated on-site longer than 90 days. All hazardous waste containers shall be stored in a secured storage area. The following requirements for the hazardous waste storage area must be implemented:

- Proper hazardous waste signs shall be posted as required by any state or federal statutes that may govern the labelling of waste;
- Secondary containment to contain spills;
- Spill containment equipment must be available;
- Fire extinguisher;
- Adequate aisle space for unobstructed movement of personnel.

6.4.3 Weekly storage area inspections shall be performed and documented to ensure compliance with these requirements. Throughout the project, an inventory shall be maintained to itemize the type and quantity of the waste generated.

6.5 Waste Disposal

6.5.1 Solid, liquid, and PPE waste will be characterized for disposal through the use of client knowledge, laboratory analytical data created from soil or groundwater samples gathered during the field activities, and/or composite samples from individual containers.

6.5.2 All waste generated during field activities will be stored, transported, and disposed of according to applicable state, federal, and local regulations. All wastes classified as hazardous will be disposed of at a licensed treatment storage and disposal facility or managed in other approved manners.

6.5.3 In general, waste disposal should be carefully coordinated with the facility receiving the waste. Facilities receiving waste have specific requirements that vary even for non-hazardous waste, so characterization should be conducted to support both applicable regulations and facility requirements.

6.6 Regulatory Requirements

The following federal and state regulations shall be used as resources for determining waste characteristics and requirements for waste storage, transportation, and disposal:

- Code of Federal Regulations (CFR), Title 40, Part 261;
- CFR, Title 49, Parts 172, 173, 178, and 179.

6.7 Waste Transport

A state-certified hazardous waste hauler shall transport all wastes classified as hazardous. Typically, the facility receiving any waste can coordinate a hauler to transport the waste. Shipped hazardous waste shall be disposed of in accordance with all RCRA/USEPA requirements. All waste manifests or bills of lading will be signed either by the client or the client's designee.

7.0 Quality Control and Assurance

7.1 Management of IDW must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 Maintain records as required by implanting the procedures in this SOP.

8.2 Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of Energy, United States (DOE). 1994. *The Off-Site Rule*. EH-231-020/0194. Office of Environmental Guidance. March.

1999. *Management of Remediation Waste under the Resource Conservation and Recovery Act (RCRA)*. Office of Environmental Policy and Assistance. 20 December.

DoD Environmental Field Sampling Handbook Revision 1.0, April 2013

Environmental Protection Agency, United States (EPA). 1991. *Management of Investigative-Derived Wastes During Site Inspections*. Office of Emergency and Remedial Response. EPA/540/G-91/009. May.

1992a. *Guidance for Performing Site Inspections under CERCLA*. EPA/540/R-92/021. Office of Emergency and Remedial Response. September.

1992b. *Guide to Management of Investigative-Derived Wastes*. Quick reference fact sheet. OSWER Dir. 9345.3-03FS. Office of Solid Waste and Emergency Response. January.

1997a. *Sending Wastes Off Site? OSC and RPM Responsibilities under the Off-Site Rule*. EPA/540-F-97-006, Office of Solid Waste and Emergency Response. September.

1997b. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IIIA. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

1998. *Management of Remediation Waste under RCRA*. EPA/530-F-98-026. Office of Solid Waste and Emergency Response. October.

(No Date). *Compliance with the Off-Site Rule During Removal Actions.* Office of Regional Counsel (Region 3). Hendershot, Michael.

Equipment Decontamination

Procedure 3-06

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes methods of equipment decontamination, to be used for activities where samples for chemical analysis are collected or where equipment will need to be cleaned before leaving the site or before use in subsequent activities.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

It is the responsibility of the **Site Safety Officer (SSO)** to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the washing area for both personnel and equipment to minimize the spread of contamination into the clean zone. Typically, for equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing cleaning solvents as described in this procedure or the Delivery Order (DO) Work Plan (WP) and distilled water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment, such as augers, pump drop pipe, and vehicles, may be accomplished using a variety of techniques.

All **Field Personnel** responsible for equipment decontamination must adhere to the site-specific health and safety plan (HSP) and must wear the personal protective equipment (PPE) specified in the site-specific HSP. Generally this includes, at a minimum, Tyvek® coveralls, steel-toed boots with boot covers or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). Air monitoring by the **SSO** may result in an upgrade to the use of respirators and cartridges in the decontamination area; therefore, this equipment must be available on site. If safe alternatives are not achievable, discontinue site activities immediately.

In addition to the aforementioned precautions, the following sections describe safe work practices that will be employed.

2.1 **Chemical Hazards associated with Equipment Decontamination**

- Avoid skin contact with and/or incidental ingestion of decontamination solutions and water.
- Utilize PPE as specified in the site-specific HSP to maximize splash protection.
- Refer to material safety data sheets, safety personnel, and/or consult sampling personnel regarding appropriate safety measures (i.e., handling, PPE including skin and respiratory).
- Take the necessary precautions when handling detergents and reagents.

2.2 **Physical Hazards associated with Equipment Decontamination**

- To avoid possible back strain, it is recommended to raise the decontamination area 1 to 2 feet above ground level.
- To avoid heat stress, over exertion, and exhaustion, it is recommended to rotate equipment decontamination among all site personnel.
- Take necessary precautions when handling field sampling equipment.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **DO Manager** is responsible for ensuring that decontamination activities comply with this procedure. The **DO Manager** is responsible for ensuring that all personnel involved in equipment decontamination shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all field equipment is decontaminated according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Procedure

Decontamination of equipment used in soil/sediment sampling, groundwater monitoring, well drilling and well development, as well as equipment used to sample groundwater, surface water, sediment, waste, wipe, asbestos, and unsaturated zone, is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- Location where the decontamination procedures will be conducted
- Types of equipment requiring decontamination
- Frequency of equipment decontamination
- Cleaning technique and types of cleaning solutions appropriate to the contaminants of concern
- Method for containing the residual contaminants and wash water from the decontamination process
- Use of a quality control measure to determine the effectiveness of the decontamination procedure

The following subsections describe standards for decontamination, including the frequency of decontamination, cleaning solutions and techniques, containment of residual contaminants and cleaning solutions, and effectiveness.

5.1 **Decontamination Area**

Select an appropriate location for the decontamination area at a site based on the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. Locate the decontamination area an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

5.2 **Types of Equipment**

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel cable. Decontamination of monitoring well development and groundwater sampling equipment includes submersible pumps, bailers, interface probes, water level meters, bladder pumps, airlift pumps, peristaltic pumps, and lysimeters. Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels, hand augers, slide hammer samplers, shovels, stainless-steel spoons and bowls, soil sample liners and caps, wipe sampling templates, composite liquid waste samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

5.3 **Frequency of Equipment Decontamination**

Decontaminate down-hole drilling equipment and equipment used in monitoring well development and purging prior to initial use and between each borehole or well. Down-hole drilling equipment, however, may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, decontaminate the drilling tools prior to drilling deeper. Initiate groundwater sampling by sampling groundwater from the monitoring well where the least contamination is suspected. Decontaminate groundwater, surface water, and soil sampling devices prior to initial use

and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

5.4 Cleaning Solutions and Techniques

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment, such as drill bits, augers, drill string, and pump drop-pipe, is steam cleaning. To steam clean, use a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, thoroughly steam wash equipment and rinse it with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a non-phosphate detergent (Alconox®, Liquinox®, or other suitable detergent) and potable water solution; (2) rinse with potable water; (3) spray with laboratory-grade isopropyl alcohol; (4) rinse with deionized or distilled water; and (5) spray with deionized or distilled water. If possible, disassemble equipment prior to cleaning. Add a second wash at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. Decontaminate these pumps by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. Decontaminate the internal surfaces by recirculating fluids through the pump while it is operating. This recirculation may be done using a relatively long (typically 4 feet) large-diameter pipe (4-inch or greater) equipped with a bottom cap. Fill the pipe with the decontamination fluids, place the pump within the capped pipe, and operate the pump while recirculating the fluids back into the pipe. The decontamination sequence shall include: (1) detergent and potable water; (2) potable water rinse; (3) potable water rinse; and (4) deionized water rinse. Change the decontamination fluids after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent; however, if samples are also to be analyzed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of distilled or deionized water. Steam-distilled water shall not be used in the decontamination process as this type of water usually contains elevated concentrations of metals. Decontamination solvents to be used during field activities will be specified in the DO WP.

Rinse equipment used for measuring field parameters, such as pH (indicates the hydrogen ion concentration – acidity or basicity), temperature, specific conductivity, and turbidity with deionized or distilled water after each measurement. Also wash new, unused soil sample liners and caps with a fresh detergent solution and rinse them with potable water followed by distilled or deionized water to remove any dirt or cutting oils that might be on them prior to use.

5.5 Containment of Residual Contaminants and Cleaning Solutions

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment, such as drill rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, use plastic sheeting and blocks or other objects to create a bermed area for collection of equipment decontamination water. Situate items, such as auger flights, which can be placed on metal stands or other similar equipment, on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Store clean equipment in a separate location to prevent recontamination. Collect decontamination fluids contained within the bermed area and store them in secured containers as described below.

Use wash buckets or tubs to catch fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices. Collect the decontamination fluids and store them on site in secured containers, such as U.S. Department of Transportation-approved drums, until their disposition is determined by laboratory analytical results. Label containers in accordance with Procedure 3-05, *IDW Management*.

6.0 Quality Control and Assurance

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment blank samples or wipe testing. Equipment blanks consist of analyte-free water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a cloth over the surface of the equipment after cleaning. These quality control measures provide "after-the fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

7.0 Records, Data Analysis, Calculations

Any project where sampling and analysis is performed shall be executed in accordance with an approved sampling and analysis plan. This procedure may be incorporated by reference or may be incorporated with modifications described in the plan.

Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

8.2 *DoD Environmental Field Sampling Handbook Revision 1.0*, April 2013

8.3 Procedure 3-05, *IDW Management*.

Surface Water Sampling

Procedure 3-10

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) for use in sampling surface water. This SOP describes the equipment, field procedures, materials, and documentation procedures necessary to collect surface water samples from shallow and deep water using a variety of samplers. The procedure and equipment required for the measurement of surface water flow velocity in association with stream gauging is also described. Specific information regarding surface water sampling and stream gauging locations and project objectives can be found in the associated Sampling and Analysis Plan (SAP).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from AECOM-Tidewater JV, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first surface water sampling location. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.

2.4 The health and safety considerations for the work associated with surface water sampling include:

- Proper selection of personal protective equipment for work around water bodies (e.g., personal flotation devices [PFDs]), as specified in the project-specific HASP.
- Appropriate health and safety protocols for working in a boat (if applicable), as specified in the project-specific HASP.

- Proper lifting techniques when retrieving surface water samplers, large muscles of the legs should be used, not the back.
- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
- To avoid slip/trip/fall hazards as a result of working on wet surfaces, wear work boots/work boot covers with textured soles.
- While wading is the preferred method for accurate flow measurement, there are obvious safety considerations that limit the flows at which wading can be accomplished. The USGS rule of thumb should e followed, which prohibits wading if the product of depth (in feet) and velocity (in feet /second) exceeds 8.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended), and in cases of extreme cold, wear fitted insulated clothing

3.0 Terms and Definitions

None.

4.0 Interferences

None.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The **Delivery Order (DO) Manager** is responsible for ensuring that surface water sampling activities comply with this procedure. The DO Manager or designee shall review all surface water sampling forms on a minimum monthly basis. The DO Manager is responsible for ensuring that all field sampling personnel involved in surface water sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure. Minimum qualifications for field sampling personnel require that one individual on the field team shall have a minimum of 6 months of experience with surface water sampling.

5.2.5 The **field sampler and/or task manager** is responsible for directly supervising the surface water sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the Program Quality Manager and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Work Plan
- Maps/Plot plan
- Tape measure, marking line or cable

- Survey stakes, flags, or buoys
- Camera and film
- Stainless steel, plastic, or other appropriate composition (e.g., Teflon) bucket
- Laboratory supplied sampling containers
- Ziploc plastic bags for samples, and sample jars
- Logbook
- Labels
- Chain of Custody (COC) forms
- Site description forms
- Cooler(s)
- Ice
- Equipment/Apparatus
- Decontamination supplies/equipment
- Spade or shovel
- Spatula
- Scoop
- Trowel
- Task specific surface water sampling equipment
- Water flow meter (Global Water Flow Probe Model FP111 or similar)

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Selection of Sampling Techniques

Proper selection of sampling points and collection methodology are essential to meeting the objectives of a surface water sampling program. Sampling points should be selected for collection of surface water samples on the basis of characteristics of the body of surface water body to be monitored, the location of the body of surface water, and its hydrologic boundaries with respect to the site. Other considerations include the contaminants of concern, logistical considerations, such as access to the surface water body, the direction of flow, and determination of a background location.

Methods of collecting surface water samples vary from hand sampling procedures at a single point to sophisticated, multipoint sampling techniques. The number and type of samples to be collected depends on the characteristics of the body of water, the amount of suspended sediment that a moving body carries, the size of the discharge area at the site, and other factors. Multipoint sampling techniques apply to larger bodies of water; the samples are composited to provide a more representative sample.

Whenever possible, the sampling device, either disposable or constructed of a nonreactive material, should hold at least 500 milliliters to minimize the number of times the liquid must be disturbed, thus reducing agitation of any sediment layers. A 1-liter polypropylene or stainless steel beaker with a pour spout and handle works well. Any sampling device might contribute contaminants to a sample. The correct sampling device will not compromise the integrity of the sample and will give the desired analytical results.

8.1.1 Shallow Water Body Surface Water Sample Collection

A dip or grab sample is appropriate for a small body of water, or for collecting near-surface samples in a larger surface water body. The sampling method involves filling a sample container by submerging it either just below the surface, or by lowering the container to a desired depth by using a weighted holder. For shallow bodies of surface water, hold the sample container carefully just beneath the water surface to avoid disturbing the streambed and stirring the sediment. Position the container's mouth so that it faces upstream, while the sampling personnel are standing downstream. Any preservative added to the sample should be added after sample collection to avoid loss of preservative. Alternatively, a transfer device may be dipped into the water, and then the contents transferred to the appropriate container containing the preservative. For near-surface sample collection in a large surface water body, a pond sampler may be used if an extended reach is required to collect a representative sample. A pond sampler consists of a single use sample container attached to a telescoping, heavy-duty, aluminium pole via an adjustable clamp attached to the end. The collection technique for shallow surface water samples can be used for near-surface samples in a large surface water body.

8.1.2 Deep Surface Water Sample Collection

For deeper surface water bodies, either sample containers or transfer devices may be used to collect a sample. A weighted holder that allows either a sample transfer device or a sample container to be lowered, opened for filling, closed, and returned to the surface is suggested for sampling deeper surface water bodies. This is because concentrations of constituents near the surface of a deeper body of surface water might differ from the total concentration distributed throughout the water column cross section and thus a surface sample would not be representative of the water body. An open container that is lowered and raised to the surface at a uniform rate so that the bottle is just filled on reaching the surface is appropriate for deeper stagnant water bodies, however this method does not collect a truly representative sample in deeper flowing surface water bodies.

Kemmerer Samplers. Collect samples near the shore unless sampling from a boat is feasible and permitted. If a boat is used, the body of water should be cross-sectioned and samples should be collected at various depths across the water in accordance with the project specific SAP. The Kemmerer Sampler consists of a glass, plastic, or Teflon bottle, a weighted sinker, a bottle stopper, and a line that is used to open the bottle and to lower and raise the sampler during sampling. The general procedure for using the sampler is as follows (or refer to manufacturer’s instructions):

1. Obtain the sampler and check the knot at the bottom of the sampler for tightness and size. The knot should be sufficiently large so that it will not pull through the central tube of the sampler.
2. Assemble the weighted bottle sampler for making the cast by pulling the trip head into the trip plate. This can be done by holding the top and bottom stoppers and giving a short, hard pull to the bottom stopper.
3. Measure and mark the desired depth on the sampling line. Tie the free end of the line to the railing of the vessel to prevent accidental dropping of the sampler.
4. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely.
5. Pull out the stopper with a sharp jerk of the sampler line or by lowering a messenger down the line to trip the stoppers.
6. Allow the bottle to fill completely, as evidenced by the cessation of air bubbles.
7. Raise the sampler and cap the bottle. Untie the line from the railing and carry the sampler to your sampling station.
8. Transfer water into appropriate sample containers. Preserve the sample, if necessary, following guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.
9. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.
10. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.
11. Immediately place the properly labeled sample bottle(s) in a cooler with ice.

12. Wipe the sample clean and decontaminate if necessary for the collection of additional samples. Decontaminate according to the procedures in SOP 3-06 Equipment Decontamination.

13. Always store the sampler in the open position (stoppers not in the tube).

Teflon Bailers. Teflon bailers can also been used to collect samples in deep bodies of water. When the use of Teflon bailers is deemed appropriate for sampling water from a specific depth, the bailers shall be equipped with a check valve that closes during sample retrieval.

1. Attach a line that is premeasured to the appropriate sampling depth to the dedicated Teflon bailer and lower to the desired depth.

2. Ensure that the check valve is engaged tugging on the line with a sharp jerk.

3. Raise the bailer and transfer the water to sample containers. Preserve the sample, if necessary, following guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.

4. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.

5. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.

6. Immediately place the properly labeled sample bottle(s) in a cooler with ice.

7. A new dedicated bailer and new line should be used for each sampling location.

Peristaltic Pump. Another method of extending the reach of sampling efforts is to use a small peristaltic pump. In this method, the sample is drawn through heavy-wall Teflon tubing and pumped directly into the sample container. This system allows the operator to reach into the liquid body, sample from depth, or sweep the width of narrow streams.

If medical-grade silicon tubing is used in the peristaltic pump, the system is suitable for sampling almost any analyte, including most organics. Some volatile stripping may occur; due to the relatively high flow rate of the pump. Therefore, avoid pumping methods for sampling volatile organics. Battery-operated peristaltic pumps are available and can be easily carried by hand or with a shoulder sling, as needed. It is necessary in most situations to change both the Teflon suction line and the silicon pump tubing between sampling locations to avoid cross contamination. This action requires maintaining a sufficiently large stock of material to avoid having to clean the tubing in the field.

Peristaltic pumps work especially well for sampling large bodies of water when a near-surface sample will not sufficiently characterize the body as a whole. When sampling a liquid stream that exhibits a considerable flow rate, it may be necessary to weight the bottom of the suction line.

Use the following procedures for collecting samples using peristaltic pumps:

1. Install clean, silicone tubing in the pump head, per the manufacturer's instructions. Pharmaceutical-grade silicone tubing (e.g., PharMed tubing) may be required for some projects depending on the analyses required. Refer to the project specific SAP for specific tubing requirements. Allow sufficient tubing on the discharge side to facilitate convenient dispensation of liquid into sample bottles but only enough on the suction end for attachment to the intake line. This practice will minimize sample contact with the silicone pump tubing. (Some types of thinner Teflon tubing may be used.).

2. Select the length of suction intake tubing necessary to reach the required sample depth and attach it to the tubing on the intake side of the pump. If necessary, a small weight composed of inert material (e.g., stainless steel) which will not react with chemicals of concern may be used to weight the intake tubing. Heavy-wall Teflon of a diameter equal to the required pump tubing will suit most applications. (A heavier wall will allow for a slightly greater lateral reach.)

3. A purge volume that is at a minimum equal to the tubing volume should be passed through the system prior to sample collection. Collect this purge volume in a bucket. Once the sample has been collected, the purged water volume can be returned to the water body.

4. Fill necessary sample bottles by allowing pump discharge to flow gently down the side of bottle with smooth laminar flow and minimal entry turbulence. Cap each bottle as it is filled.
5. Preserve the sample, if necessary, following guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.
6. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.
7. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.
8. Immediately place the properly labeled sample bottle in a cooler with ice.
9. Allow the system to drain thoroughly, and then disassemble.

8.2 Transfer Devices

Samples from various locations and depths can be composited if project quality objectives indicate that it is appropriate; otherwise, collect separate samples. Identify approximate sampling points on a sketch of the water body. Use the following procedures for collecting samples using transfer devices:

1. Submerge a stainless steel dipper or other suitable device, causing minimal disturbance to the surface of the water and the sediment at the floor of the surface water body. Note the approximate depth and location of the sample source (e.g., 1 foot up from bottom or just below the surface).
2. Allow the device to fill slowly and continuously.
3. Retrieve the dipper or device from the surface water with minimal disturbance.
4. Remove the cap from the sample bottle and slightly tilt the mouth of the bottle below the dipper or device edge.
5. Empty the dipper or device slowly, allowing the sample stream to flow gently down the side of the bottle with smooth laminar flow and minimal entry turbulence.
6. Continue delivery of the sample until the bottle is filled.
7. If necessary, preserve the sample according to guidelines in the project-specific SAP. In most cases, place preservatives in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.
8. Check that a Teflon liner is present in the cap, if required. Secure the cap tightly.
9. Fill out the sample label and record all relevant information in the sample collection form, the field logbook, and/or the field laptop/tablet. In addition, the chain of custody form should be filled out as soon as possible. These procedures should be done in accordance with SOP 3-03 Recordkeeping, Sample Labeling, and Chain of Custody.
10. Dismantle the sampler and decontaminate according to the procedures in SOP 3-06 Equipment Decontamination.

Multipoint sampling techniques that represent both dissolved and suspended constituents and both vertical and horizontal distributions are applicable to larger bodies of water. Subsequent to sample collection, multipoint sampling techniques may require a compositing and sub-sampling process to homogenize all the individual samples into the number of subsamples required to perform the analyses of interest. Homogenizing samples is discouraged for samples collected for volatile organic analysis, because aeration causes a loss of volatile compounds. If collection of composite samples is required, then include the procedure for compositing in the project-specific work plan.

The sampling devices selected must not compromise sample integrity. Collect samples with either disposable devices, or devices constructed of a nonreactive material, such as glass, stainless steel, or Teflon. The device must have adequate capacity to minimize the number of times the liquid must be disturbed, reducing agitation of any sediment layers. Further, the device must be able to transfer the water sample into the sample container without loss of volatile compounds. A single- or double-check valve or stainless steel bailer made of Teflon equipped with a bottom discharging device may be utilized.

All equipment used for sample collection must be decontaminated before and after use in accordance with Procedure 3-06 – Equipment Decontamination.

8.3 Flow Velocity Measurement

Surface water flow velocity measurement is required to conduct stream gauging for the purpose of determining discharge volumes and associated potential generation, transport and delivery of point and non-point source pollutants. Gauging is the process of measuring stream water flow, which has the equation Q=V*A, where Q = flow, V = stream water velocity and A = stream cross sectional area. Measuring flow, Q, directly is challenging so stream gauging is typically done by measuring both the water velocity (V), and the cross sectional area (A) at the monitoring site. Measuring water velocity can be done in several ways with the most popular being the use of a water velocity or current meter, These meters provide the water velocity measurement, but require the user to measure the cross sectional area manually to calculate the stream discharge measurement. Use the following procedure to measure flow and discharge in a small, wadable stream:

1. Select a location of the stream with a straight reach, reasonably free of large rocks or obstructions, with a relatively flat streambed, away from the influence of abrupt changes in channel width.
2. Determine the width of the stream and string a cable or measuring tape across the stream at a right-angle to the flow. Divide the width into 20 to 25 equal segments (streams less than 10 feet wide may not allow for as many segments) using tape or string to mark the center of each segment so that each one has no more than 10 percent of the total streamflow.
3. Measure the depth from the water surface to the bottom at each measuring point using a graduated rod (e.g., the depth marking on the outside of the meter staff).
4. At each mark, measure the velocity of the water (see below). Where depth is less than 2.5 feet, take a single velocity measurement at 0.6 of the total depth below the water surface. For depths of 2.5 feet or more, the average of velocity measurement taken at 0.2 and 0.8 of depth should be taken.
5. Measure flow by placing the housing of the flow probe propeller at the measuring point with the arrow on the side of the housing in the direction of the flow (i.e., pointing downstream).
6. Reset the unit and measure the velocity for a period of 40 seconds. At the end of the recording period, record the resulting average velocity (in feet/second).
7. Calculate the stream discharge for each segment by multiplying the width of the segment and the measured depth (giving area) times the velocity measured in that segment.
8. Calculate total stream discharge by summing all of the segment discharges

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality Control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation, holding times, container types, as well as various QC samples such as trip blanks, field blanks, equipment blanks, and field duplicates.

10.0 Data and Records Management

10.1 Field notes will be kept during sampling activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. During the completion of sampling activities, fill out the sample logbook and transmit forms to the DO Manager for storage in project files.

10.2 Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-

900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

DoD Environmental Field Sampling Handbook, Revision 1.0. April 2013.

Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. EPA/540/P-87/001, EPA, Office of Emergency and Remedial Response, Washington, D.C.

Surface Water Flow Measurement for Water Quality Monitoring Projects, National Nonpoint Source Monitoring Program, Tech Notes 3. March 2008.
https://www.bae.ncsu.edu/programs/extension/wqg/319monitoring/TechNotes/technote3_surface_flow.pdf

USGS Techniques of Water Resource Investigation
http://pubs.usgs.gov/twri

Monitoring Well Installation

Procedure 3-12

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the methods to be used during the installation of groundwater monitoring wells. It describes the components of monitoring well design and installation and sets forth the rationale for use of various well installation techniques in specific situations.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Delivery Order (DO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.

2.2 Before well installation commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated well locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

2.3 Physical Hazards Associated with Well Installation

- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
- When using an approved retractable-blade knife, always cut away from one self and make sure there are no other people in the cutting path or the retractable-blade knife.
- To avoid slip/trip/fall conditions during drilling activities, keep the area clear of excess soil cuttings and groundwater. Use textured boots/boot cover bottoms in muddy areas.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and personal protective equipment (PPE), drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- To avoid hazards associated with subsurface utilities, ensure all sampling locations have been properly surveyed as described in SOP 3-01, Utility Clearance.
- Be aware of restricted mobility caused by PPE.

3.0 Terms and Definitions

3.1 **Annulus:** The annulus is the down-hole space between the borehole wall and the well casing and screen.

3.2 **Bridge:** A bridge is an obstruction in the drill hole or annulus. A bridge is usually formed by caving of the wall of the well bore, by the intrusion of a large boulder, or by the placement of filter pack materials during well completion. Bridging can also occur in the formation during well development.

3.3 **Filter Pack:** Filter pack is sand or gravel that is smooth, uniform, clean, well-rounded, and siliceous. It is placed in the annulus of the well between the borehole wall and the well screen to prevent formation materials from entering the well and to stabilize the adjacent formation.

3.4 **Grout:** Grout is a fluid mixture of cement and water that can be forced through a tremie pipe and emplaced in the annular space between the borehole and casing to form an impermeable seal. Various additives, such as sand, bentonite, and polymers, may be included in the mixture to meet certain requirements.

3.5 **Heaving (Running) Sands:** Loose sands in a confined water-bearing zone or aquifer which tend to rise up into the drill stem when the confining unit is breached by the drill bit. Heaving sands occur when the water in

the aquifer has a pressure head great enough to cause upward flow into the drill stem with enough velocity to overcome the weight of the sand.

3.6 **Sieve Analysis:** Sieve analysis is the evaluation of the particle-size distribution of a soil, sediment, or rock by measuring the percentage of the particles that will pass through standard sieves of various sizes.

4.0 Interferences

4.1 Heaving sands may be problematic in unconsolidated sands encountered below the water table.

4.2 Rotary drilling methods requiring bentonite-based drilling fluids should be used with caution to drill boreholes that will be used for monitoring well installation. The bentonite mud builds up on the borehole walls as a filter cake and permeates the adjacent formation, potentially reducing the permeability of the material adjacent to the well screen.

4.3 If water or other drilling fluids have been introduced into the boring during drilling or well installation, samples of these fluids should be obtained and analyzed for chemical constituents that may be of interest at the site. In addition, an attempt should be made to recover the quantity of fluid or water that was introduced, either by flushing the borehole prior to well installation and/or by overpumping the well during development.

4.4 Track-mounted drill rigs are suitable for travelling on many types of landscapes that truck-mounted units cannot access, but may have limitations on extremely uneven or soft terrain.

4.5 Care should be taken to prevent cross-contamination between well locations. All drilling equipment coming in contact with potentially contaminated soil and/or groundwater will be decontaminated by the drilling subcontractor prior to initial drilling activities and between drilling locations in accordance with SOP 3-06, Equipment Decontamination.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 **Delivery Order (DO) Managers** are responsible for issuing sampling and analysis plans (SAPs) that reflect the procedures and specifications presented in this procedure. Individual municipalities, county agencies, and possibly state regulatory agencies enforce regulations that may include well construction and installation requirements. The **DO Manager** shall be familiar with current local and state regulations, and ensure that these regulations are followed. The **DO Manager** is responsible for ensuring that all personnel involved in monitoring well installation shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for direct supervision of the installation of monitoring wells and ensuring that procedures and specifications are implemented in the field in accordance with the approved SAP and well installation permits. The qualifications for the **Field Manager** must be in accordance with local jurisdictions with authority over the operations conducted.

5.2.4 All field personnel are responsible for the implementation of this procedure.

5.2.5 The on-site hydrogeologist/engineer is expected to obtain a description of the lithologic samples obtained during the excavation and construction of a monitoring well. These data are often required to provide guidance regarding the installation of specific components of the monitoring well. Guidance for lithologic sample collection and sample description is contained within SOP 3-16, Soil and Rock Classification.

6.0 Equipment and Supplies

6.1 Materials provided by the drilling contractor may include:

- Drill rig, drill rods, hollow stem augers, etc.
- Decontamination equipment (e.g., steam cleaner, high-pressure washer, brushes, etc.)
- Decontamination pad materials
- Well screen/riser pipe with flush-threaded couplings including riser and bottom caps
- Clean, filter sand
- Bentonite chips or pellets
- Cement grout and tremie pipe
- Portland cement for well pad completion
- Steel protective riser covers and locking caps
- Weighted calibrated tape
- Split-spoon samplers
- 55-gallon drums or containers for drill cuttings, decontamination fluids, etc.

6.2 In addition to those materials provided by the drilling contractor, equipment and materials required by the project geologist/engineer may include, but is not limited to, the following:

- Photoionization Detector (PID)
- Spill kit, including at a minimum sorbent pads and shovel (if not provided by subcontractor)
- Plastic sheeting
- Teaspoon or spatula
- Resealable plastic bags
- Boring Log Records
- Decontamination materials (per SOP No. 3-06 - Equipment Decontamination)
- Weighted measuring tape for depth measurement
- Soil logging materials (e.g. USCS classification field card, millimeter rule, hand lens, etc.)
- Survey lathes or pin flags
- Digital camera
- PPE as required by the HASP
- Planning documents including the site-specific HASP and SAP
- Large indelible ink or paint pen
- Field logbook/field forms/site maps (water proof)

7.0 Procedure

7.1 **General Procedures**

- Specific drilling, sampling, and installation equipment and methodology will be dictated by the type of well to be installed (e.g., single case (Type II), double case (Type III), bedrock, etc.), geologic characteristics of the site, the type of contaminants being monitored, and local and state regulations.
- For access to locations when travelling over difficult terrain, an appropriate line should be chosen before mobilizing the drill rig or other support vehicles. If clearing of trees or ground cover is required, perform these activities in advance to avoid down time. Avoid wet or soft areas where possible or use ground

mats and/or timbers to aid in supporting the rig as it travels. If drilling on soft material, place geomatting and ground mats under the rig tracks or stabilizers prior to drilling.

- A utility locate must be conducted to identify all underground utilities at the site prior to drilling (refer to SOP 3-01, Utility Clearance*)*. Proper clearance procedures for aboveground/overhead utilities must also be followed as specified in the HASP.
- Although new well materials (well screen and riser pipe) generally arrive at the site boxed and sealed within plastic bags, it is sometimes necessary to decontaminate the materials prior to their use. Well materials should be inspected by the project geologist/engineer upon delivery to check for cleanliness. If the well materials appear dirty, or if local or regional regulatory guidance requires decontamination, then well material decontamination should be performed by the drilling subcontractor in accordance with SOP 3-06, Equipment Decontamination.
- The diameter of the borehole must be a minimum of 2 inches greater than the outside diameter of the well screen or riser pipe used to construct the well. This is necessary so that sufficient annular space is available to install filter packs, bentonite seals, and grout seals, and allow the passage of tremie pipe where grouting at depth is required. Bedrock wells may require reaming after coring in order to provide a large enough borehole diameter for well installation.
- When soil sampling is required (refer to the SAP), soil samples will be collected for visual logging by advancing split-spoon samplers through the augers. The soil will be visually logged by a field geologist and include lithologic characteristics (i.e., soil type, color, density, moisture content, etc.) using the the methods described in SOP 3-16, Soil and Rock Classification. This information will be recorded on a boring/well log form, along with well construction details.

7.2 Drilling Techniques

Drilling of monitoring well boreholes may be accomplished by a variety of methods as described below. Preferred methods include those that temporarily case the borehole during drilling (i.e., hollow stem auger and sonic methods) using an override system. Other methods can be used where specific subsurface conditions or well design criteria dictate.

- Hollow stem auger (HSA) – Borings are advanced by rotating steel hollow stem augers with an attached cutting head. Soil cuttings are displaced by the cutting head and transported to the surface via continuous spiral flights attached to each auger stem. This method is widely used for unconsolidated soils that have a tendency to collapse within the boring. A bottom plug can be placed in the bottom auger to prevent soils from entering and clogging the auger, especially in the case of heaving sands. However, a bottom plug cannot be used when soil samples are to be collected through the augers. Soil plugs that accumulate in the bottom of the auger must be removed or knocked out prior to sampling or well installation.
- Solid stem auger – This type of drilling method is similar to HSA drilling using a solid stem or sealed hollow stem auger flights to advance the boring. Solid stem, continuous flight auger use is limited to semi-consolidated sediments or to cohesive or semi-cohesive unconsolidated sediments that don't have a tendency to collapse when disturbed.
- Sonic methods – Sonic drilling consists of advancing concentric hollow drill casings (inner and outer) using rotation in conjunction with axial vibration of the drill casing. Once the casings are advanced to the appropriate depth, the inner string is removed with a core of drill cuttings while the outer casing remains in place to keep the borehole open. Cuttings are removed from the inner casing relatively intact for logging or sampling purposes. This drilling method is used for a variety of soil types, from heaving sands to consolidated or indurated formations. Smearing of the formation along the borehole walls is minimal since moderate vibration and rotation techniques are used to advance the casings. Since the total borehole diameter in sonic drilling is only incrementally larger than the inner casing diameter, care should be taken during installation of the monitoring well to ensure the well is centered and adequate space is available for annular materials.
- Rotary methods (water or mud) – Rotary drilling methods consist of drill rods coupled to a drill bit that rotates and cuts through the soils to advance the borehole. Water or drilling fluid ("mud") is forced through the hollow drill rods and drill bit as the rods are rotated. The soil cuttings are forced up the borehole with the drilling fluids to the surface and the fluids recirculated. The drilling fluid provides a hydrostatic pressure that reduces or prevents the borehole from collapsing. Clean, potable water must be used for water-rotary drilling to prevent introducing trace contaminants. A sample of the potable

water should be collected during the course of well installation for analysis of the same parameters defined for the groundwater samples. If mud-rotary is used to advance boreholes, potable water and bentonite drilling mud should only be used. No chemical additives shall be mixed in the drilling fluid to alter viscosity or lubricating properties. Adequate well development is essential for removal of drilling mud and fluids from the formation materials and ensure collection of representative groundwater samples.

- Rotary methods (Air) – Air rotary methods are similar to water rotary but use high air velocities in place of drilling fluids to rotate the drill bit and carry the soil cuttings up the borehole to the surface. Care must be taken to ensure that contaminants are not introduced into the air stream from compressor oils, etc. Most compressor systems are compatible with a coalescing filter system. Cuttings exiting the borehole under pressure must be controlled, especially when drilling in a zone of potential contamination. This can be accomplished by using an air diverter with hose or pipe to carry the cuttings to a waste container. Letting the cuttings blow uncontrolled from the borehole is not acceptable.

7.3 **Well Construction and Installation**

- If rotary drilling techniques are used, the borehole should be flushed or blown free of material prior to well installation. If hollow stem augers are used, the soil or bottom plug should be removed and the augers raised approximately six inches above the bottom of the borehole, while slowly rotating the augers to remove cuttings from the bottom of the boring. The depth of the borehole should be confirmed with a weighted, calibrated tape.
- The riser pipe and screen should be connected with flush-threaded joints and assembled wearing clean, disposable gloves. No solvent or anti-seize compound should be used on the connections. The full length of the slotted portion of the well screen and unslotted riser pipe should be measured and these measurements recorded on a well construction form (Attachment 1).
- If placed in an open borehole, the assembled well should be carefully lowered and centered in the borehole so that the well is true, straight, and vertical throughout. Centering can also be accomplished with the use of centralizers, if necessary. However, centralizers should be placed so that they do not inhibit the installation of filter sand, bentonite seal, and annular grout. Wells less than 50 deep generally do not require centralizers.
- If hollow stem augers are used, the well should be lowered through the augers and each auger flight removed incrementally as the filter sand, bentonite seal, and grout are tremmied or poured into the annular space of the well. The well should be temporarily capped before filter sand and other annular materials are installed.
- Clean, silica sand should be placed around the well screen to at least 1 foot above the top of the screen. The filter sand should be appropriately graded and compatible with the selected screen size and surrounding formation materials. In general, the filter pack should not extend more than 3 feet above the top of the screen to limit the thickness of the monitoring zone. As the filter pack is placed, a weighted tape should be lowered in the annular space to verify the depth to the top of the layer. This measurement will be recorded on the well construction form (Attachment 1). If necessary, to eliminate possible bridging or creation of voids, placement of the sand pack may require the use of a tremie pipe. Tremie pipe sandpack installations are generally suggested for deeper wells and for wells which are screened some distance beneath the water table.
- A minimum 2-foot thick layer of bentonite pellets or slurry seal will be installed immediately above the filter sand to prevent vertical flow within the boring from affecting the screened interval. Bentonite chips/pellets must be hydrated if place above the water table prior to grouting. If bridging is of concern as in the case of deep wells, powdered bentonite may be mixed with water into a very thick slurry and a tremie pipe used to place the seal to the desired depth. Placement of the bentonite seal in the borehole will be recorded on the well construction form (Attachment 1).
- The remaining annular space around the well will be grouted from the top of the bentonite seal to the surface with a grout composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.
- Each well riser will be secured with an expandable, locking cap (vented if possible). Optionally, a hole can be drilled in the upper portion of the riser to allow venting of the well.
- The well will be completed within a concrete well pad consisting of a Portland cement/sand mixture. Well pads are generally 3 feet by 3 feet square but may be larger or smaller depending on site

conditions and state-specific well construction standards. Round concrete well pads are also acceptable. A minimum of 1 inch of the finished pad should be below grade to prevent washing and undermining by soil erosion.

- If completed as a flush-mount well, the well riser will be cut off approximately 4 to 6 inches below ground surface and an expandable, locking cap placed on the well riser. The area around the riser is dug out and a steel well vault or manhole cover placed over the riser and set almost flush to the ground to protect the well. The manhole cover should be water-tight and secured with bolts to prevent casual access. The well pad will then be constructed around the well vault and slightly mounded at the center and sloping away to prevent surface water from accumulating in the well vault.
- If completed as a stick-up well, the well riser is cut approximately 2.5 to 3 feet above the ground surface and an expandable, locking cap placed on the well riser. A steel guard pipe with hinged, locking cap is placed over the well riser as a protective casing. The bottom of the guard pipe will be set approximately 2 feet below ground surface and sealed by pouring concrete from the top of the annular grout around the pipe to grade. The concrete well pad should be completed at the same time. Weep holes will be drilled in the base of the guard pipe to facilitate draining of rainwater or purge water from inside the guard pipe.
- Bumper posts or bollards may be necessary for additional well protection, especially in high traffic areas. The bumper posts should be placed around the well pad in a configuration that provides maximum protection to the well and extend a minimum of 3 feet above the ground.

7.4 Double Cased Wells

Under certain site conditions, the use of a double-cased or telescoping (Type III) well may be necessary. Installation of double-cased wells may be required to prevent the interconnection of two separate aquifers, seal off a perched aquifer without creating a vertical hydraulic conduit, prevent cross-contamination during construction of wells in deeper aquifers hydro-stratigraphically below impacted aquifers, or case off highly impacted soils present above the aquifer to prevent potential “dragging down” of contaminants.

Similar to conventional wells, construction of double-cased wells can be accomplished using a varety of drilling methods. Well construction is initiated by “keying” a large diameter, outer casing into a stratigraphic zone of low permeability (clay layer or bedrock). The size of the outer casing should be a minimum of 2 inches greater than the outside diameter of the inner casing to allow installation of annular seal materials during well completion. A pilot borehole should be drilled through the overburden soil and/or contaminated zone into a clay confining layer or bedrock. The borehole for the outer casing should be of sufficient size to contain the outer casing with a minimum of 2 inches around the outside diameter to allow sufficient annular space for tremie or pressure grouting. The boring should extend a minimum of 2 feet into a clay layer and a minimum of 1 foot into bedrock, if possible, to ensure an adequate seal. The boring should never breach a confining layer or keyed zone under any circumstances.

Once the boring is completed, the outer casing can be set in the borehole and sealed with grout. The outer casing can be set two ways, with or without a bottom cap. If no bottom cap is applied, the casing is usually driven approximately 6 inches into the clay confining unit. A grout plug is generally placed in the bottom of the casing and once set, standing water in the casing is evacuated prior to drilling below the casing. As an alternative, a cap can be placed on the bottom of the casing and if set below the water table, the casing can be filled with clean, potable water to hold down the casing in the boring. Grouting should be conducted using tremie-grouting or pressure-grouting methods by pumping grout into the annular space between the outer casing and the borehole wall from the bottom of the casing to the ground surface. Grout around the casing should be allowed to cure at least 24 hours before attempting to drill through the bottom.

Once the grout is cured, a smaller diameter drill pipe/bit is used to bore through the grout plug or bottom cap to the desired well depth. The well is then constructed as described in Section 7.3 above.

7.5 Post Installation Procedures

- Wells should be permanently labelled or marked for identification. Well tags can be used to record the site name, well number, total depth, installation date, etc. At a minimum, the well number will be written in indelible marker or paint on both the outside of the protective casing and inside beneath the casing lid, as well as on the riser pipe.
- A measuring point will be marked on the top of the riser pipe for taking water level measurements. The measuring point can be notched using a knife or saw or can be marked with a waterproof marker or paint. The measuring point will also be the point which will be surveyed for vertical elevation data.

- Upon completion, the following measurements will be taken by the field geologist/engineer and recorded on the well construction diagram.
    - Depth to static water level
    - Depth of non-aqueous phase liquid (NAPL), if present
    - Total depth of well measured from top of casing (TOC)
    - Height of well casing above ground surface
    - Height of protective casing above ground surface
- All monitoring wells will be surveyed for horizontal and vertical control by a licensed surveyor.
- Investigation-derived waste (IDW) including drill cuttings, spent materials (e.g., PPE), and decontamination water should be properly managed in accordance with SOP 3-05, IDW Management.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the SAP. Certain quality control (QC) measures should be taken to ensure proper well installation and construction in accordance with this SOP, project specific SAP, and applicable well standards.

8.2 The borehole will be checked for total open depth, and extended by further drilling or shortened by backfilling, as required before installation of the well materials.

8.3 Water level and NAPL presence will be checked during well installation to ensure that the positions of well screen, filter sand, and seals relative to water level conform to project requirements

8.4 The depth to top of each layer of annular materials (i.e., filter sand, bentonite, grout) will be verified and adjusted as necessary for proper placement.

9.0 Records, Data Analysis, Calculations

All field information will be recorded in the field logbook and/or standardized field forms by field personnel. Field data recorded will include drilling contractor information, drilling methods, well material and construction information provided on the boring logs and well construction forms, observations or problems encountered during drilling, fluid level data, and any deviations from the procedures in this SOP and other project plans. Well Construction Forms (Attachment 1) will provide visual and descriptive information the monitoring well and are often the most critical form of documentation generated during the installation of a monitoring well. The field logbook is kept as a general log of activities and should not be used in place of the boring log.

10.0 Attachments or References

10.1 Attachment 1 – Monitoring Well Construction Form

10.2 Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. Office of Solid Waste and Emergency Response. EPA/540/P-87/001.

10.3 EPA. 1990. *Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells*. EPA/600/4-89/034. Office of Research and Development, Washington. March.

10.4 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

10.5 EPA, 2008. SESD Operating Procedure SESDGUID-101-R0: *Design and Installation of Monitoring Wells*. USEPA, Science and Ecosystem Support Division (SESD), Athens, Georgia. Effective Date February 18, 2008.

10.6 U.S. Army Corps of Engineers. 2008. Manual No. EM 385-1-1. *Safety and Health Requirements*. 15 November 2008. http://140.194.76.129/publications/eng-manuals/em385-1-1/2008_English/toc.html.

10.7 SOP 3-01, *Utility Clearance*.

10.8 SOP 3-05, *IDW Management*

10.9 SOP 3-06, *Equipment Decontamination*.

10.10 SOP 3-16, *Soil and Rock Classification.*

Attachment 1
Monitoring Well Construction Form

Monitoring Well Construction Log Form

Project Name: _____ **Well No.** _____

Project Location: _____ **Project No.** _____

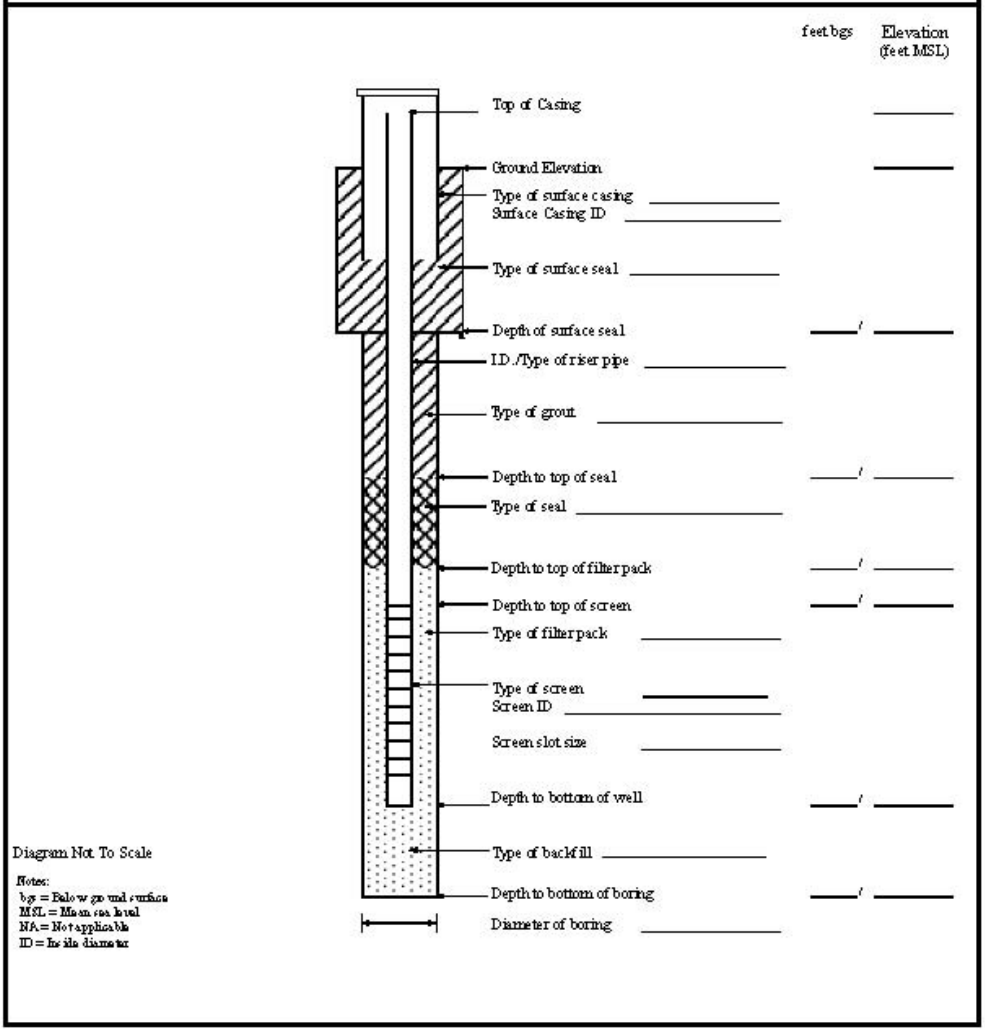
Installed By: _____ **Observed By:** _____ **Date of Well Completion** _____

Method of Installation: _____

Well Type (circle one): **Single Cased** **Double Cased**

Coordinates: **Northing** _____ **Survey Datums:** _____

Easting _____ _____



Monitoring Well Development

Procedure 3-13

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures used for developing newly installed monitoring wells and/or redeveloping existing wells.

1.2 The purpose of well development is to remove interferences from a well to provide better connection between the well and the formation, to improve pumping performance of the well, and to be able to collect more representative information from the well (e.g., samples, test results, etc.). Proper well development will:

- Remove drilling residuals (e.g., water, mud) from the borehole and surrounding formations;
- Improve or restore hydraulic conductivity of the surrounding formations which may have been disturbed during the drilling process;
- Remove residual fines from the well screen and sand pack (filter pack) materials, thus reducing turbidity of groundwater and permitting the collection of more representative groundwater samples.

1.3 There may be circumstances where well development is not desirable, for example, in the presence of non-aqueous phase liquids (NAPL) or other significant contamination if development could worsen the contaminant impact. If NAPL begins to intrude during development, the development process will be halted. This situation will be considered a cause for sample modification requiring approval by the Delivery Order (DO) Manager and other stakeholders, as applicable.

1.4 The applicable well development procedures for a particular site may be subject to State or local regulatory requirements. In all cases, the project team should consult their local regulatory requirements and document the selected well development procedure in the project-specific Sampling and Analysis Plan (SAP). For project-specific information refer to the SAP, which takes precedence over these procedures.

1.5 This procedure is the Program-approved professional guidance for work performed by AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV).

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the DO SAP and/or direction from the Site Safety Officer (SSO).

2.2 Monitoring well development may involve chemical hazards associated with potential contaminants in the soil or aquifer being characterized and may involve physical hazards associated with use of well development equipment.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Equipment/materials used for development may react with the groundwater during development. Appropriate development equipment has been selected for the anticipated condition of the groundwater.

4.2 Appropriate development methods such as using a surge-block to flush suspended fines in the groundwater in and out of the well screen can improve the yield of wells and improve their potential to be

developed successfully. However, the effectiveness of development can be significantly reduced in wells that do not yield sufficient water to allow this flushing to take place.

4.3 For formations with a significant content of fine-grained materials (silts and clays), or wells with improperly sized screens, it may not be possible to reduce turbidity to commonly acceptable levels. Possible solutions may include collecting a sample even if excessively turbid, or installing a replacement well.

4.4 Development itself disturbs the surrounding formation and disrupts equilibrium conditions within the well. Groundwater samples will not be collected until a minimum of 24 hours after a well is developed to allow conditions to stabilize. For sites with fine-grained formations (silts and clays) and highly sorptive contamination, a longer time period between development and sampling should be considered.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **DO Manager** is responsible for ensuring that well development activities comply with this procedure. The **DO Manager** is responsible for ensuring that all personnel involved in well development shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for ensuring that all well development activities are conducted according to the either this procedure or the applicable procedure presented in the project-specific SAP.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the well development procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 This equipment list was developed to aid in field organization and should be used in planning and preparation. Depending on the site-specific requirements and the development method selected, additional or alternative material and equipment may be necessary. In addition, for sites where groundwater is expected to be contaminated, the materials to be placed down the well and in contact with groundwater should be evaluated so that they are compatible with the chemical conditions expected in the well.

6.2 Equipment and materials used for well development may include, but is not limited to:

Well development equipment

- Surge block
- Disposable Teflon bailers, appropriate to the diameter of the well(s): 1-inch to 1.5-inch for 2-inch inside diameter (ID) monitoring wells.
- Watterra® footvalve
- Electric submersible pump
- 12-volt power source for electric pump
- High density polyethylene (HDPE) tubing appropriately sized for Watterra® footvalve and/or electric submersible pump
- Drums or containers for storage of purge water
- Nephelometer to measure turbidity
- Multi-parameter water quality meter(s) to measure temperature, pH, conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP)

- Instrument calibration solutions
- Water level meter
- Oil/water interface probe

General equipment

- Project-specific plans including the site-specific HASP and SAP
- Field notebook/field forms/site maps
- Indelible markers/pens
- 5-gallon buckets

Equipment decontamination supplies (refer to SOP 3-06, Equipment Decontamination)

- Health and safety supplies, including personal protective equipment (PPE)
- Appropriate hand tools
- Keys or combinations to access monitoring wells
- Distilled/deionized water supply
- Disposable bailer string (polypropylene)
- Plastic trash bags

7.0 Procedure

Development generally consists of removing water and entrained sediment from the well until the water is clear (to the extent feasible) and the turbidity is reduced, which indicates the well is in good hydraulic connection with the surrounding formation. In addition to simply removing water, development can be improved when flushing through the well screen and gravel pack takes place in both directions, that is, both into the well and into the formation. This action breaks down sediment bridges that can occur in the formation or sand pack, which reduce the connection between the well and the formation

7.1 General Preparation

- All down-well equipment should be decontaminated prior to use and between well locations in accordance with SOP 3-06, Equipment Decontamination
- Although equipment is decontaminated between well locations, if wells are known or suspected to be contaminated based on observations during well installation, it is recommended that well development be conducted in order from the least contaminated to the most contaminated well to minimize the chances of cross-contamination.
- Management of investigation-derived waste (IDW), including development purge water and miscellaneous expendable materials generated during the development process, will be conducted in accordance with SOP 3-05, IDW Management.
- Prior to accessing the well, the wellhead should be cleared of debris and/or standing water. Nothing from the ground surface should be allowed to enter the well.
- The depth to water and total well depth should be measured with a water level meter and recorded in the field logbook or on a Well Development Record (Attachment 1). This information will be used to calculate the volume of standing water (i.e., the well volume) within the well, and plan the specific details of the well development. If wells are suspected to contain NAPL, an oil/water interface probe should be used to measure liquid levels and depth to bottom of the well.
- Permanent monitoring wells will be developed no sooner than 24 hours after well installation is completed in order to allow well completion materials to set properly.

7.2 Monitoring Well Development Procedures

Generally, development will begin by gently surging the well with a surge block or bailer as described in Sections 7.2.1 and 7.2.2, respectively. Surging can become more vigorous as development progresses but initially the well must be gently surged to allow material blocking the screen to become suspended without damaging the well. Next, a bailer can be used to remove the sediment settled at the base of the well. A bailer, Watterra® pump, or electric submersible pump will then be used to purge the well, per Sections 7.2.2, 7.2.3, or 7.2.4, respectively. The well will be purged until the removed water becomes less turbid or per the requirements of the project-specific SAP, or State or local requirements. At this

point the well will be surged again with a surge block or bailer. The well can be surged more vigorously at this point. After surging, the well will be purged again until the turbidity once again decreases. The surge/purge cycle should be completed at least three times during the development process. After the last surge, the well will be purged until the development completion criteria outlined in 7.3.2 or per the project-specific SAP are met.

7.2.1 Surge Block

The default method of well development is the use of a surge block in conjunction with pumping or bailing to remove sediment-laden water.

- The construction of the surge block must be appropriate for the diameter of the well. The surge block must be mounted on rods or other stiff materials to extend it to the appropriate depths and to allow for the surge block to be moved up and down in the well.
- Insert the surge block into the well and lower it slowly to the screened or open interval below the static water level. Start the surge action by slowly and gently moving the surge block up and down in the well. A slow initial surging, using plunger strokes of approximately 1 meter or 3 feet, will allow material which is blocking the screen to separate and become suspended.
- After 5 to 10 plunger strokes, remove water from the well using a separate bailer (Section 7.2.2) or pumping techniques (Sections 7.2.3 or 7.2.4). The returned water should be heavily laden with suspended fines. The water will be discharged to 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- In some cases, the bailer or Watterra® foot valve can act as a surge block, flushing water in and out of the well screen as groundwater is removed.
- Repeat the process of surging and pumping/bailing. As development continues, slowly increase the depth of surging to the bottom of the well screen. Surging within the riser portion of the well is neither necessary nor effective.

7.2.2 Bailer

- Tie a string or other cable securely to the bailer. Lower it to the screened or open interval of the monitoring well below the static water level.
- The bailer may be raised and lowered repeatedly within the screened interval to attempt to simulate the action of a surge block by pulling fines through the well screen, and pushing water out into the formation to break down bridging.
- With the bailer full of water, remove it from the well and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The Watterra® system (Section 7.2.3) or electric submersible pump (Section 7.2.4) may be used as a complementary development method to the bailer, especially when removal of additional water at a faster rate is beneficial.
- Continue alternately surging and bailing, monitoring the purge water periodically (Section 7.3.1) until development completion criteria are met (Section 7.3.2).

7.2.3 Watterra® system

- Attach high-density polyethylene (HDPE) tubing to the decontaminated Watterra® pump foot valve
- Lower the foot valve and tubing assembly near the bottom of the well.
- Lift and lower the tubing to allow water to enter the Watterra® foot valve and travel up the tubing and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The lifting and lowering action of the Watterra® sysem will cause some surging action to aid in breaking up fine material in the surrounding formation.
- A bailer (Section 7.2.2) may be used as a complementary development method to the Watterra® system, especially during the initial stages of development when a high volume of sediment may be required to be removed.
- An electric submersible pump (Section 7.2.4) may also be used as a complementary development method to the Watterra® system, especially when more volume of water is desired to be pumped or the turbidity criteria cannot be met due to the surging action of the Watterra® system.

- Continue alternately surging and pumping, monitoring the purge water periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.2.4 Electric Submersible Pump

- Attach HDPE tubing to the decontaminated electric submersible pump.
- Lower the pump and tubing assembly near the bottom of the well, at least a few inches above the well total depth.
- Begin pumping, discharging the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- Continue alternately surging and pumping, monitoring the purge water discharge periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.3 Discharge Monitoring

7.3.1 Monitoring the Progress of Development

The progress of the development is evaluated through visual observation of the suspended sediment load and measurement of the turbidity and other parameters in the purged diischarge water. As development progresses, the water should become clearer, measured turbidity should decrease, and specific capacity (pumping rate divided by drawdown) should stabilize. Water quality parameters, including DO, conductivity, ORP, pH, temperature, and turbidity may be measured and recorded periodically to determine the progress of development using the criteria outlined in Section 7.3.2 or per the project-specific SAP. Water quality parameters should be measured on each well volume removed.

7.3.2 Completion of Development

The well will be considered developed when the following criteria are met or per the criteria set forth in the project-specific SAP:

- A minimum of three times the standing water volume in a well (to include the well screen and casing plus saturated annulus, assuming 30 percent porosity) is removed.
- Groundwater parameters for three consecutive standing water volumes are within the following:
    - pH – within ± 0.2 units
    - Specific conductivity – within ± 3%
    - ORP – within ± 10 mV
    - Temperature – within ±1 degree Celsius
    - Turbidity – at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU.
- The sediment thickness remaining within the well is less than 1 percent of the screen length or less than 30 millimeters (0.1 ft) for screens equal to or less than 10 feet long.

Dissolved oxygen (DO) readings may be recorded but DO readings will not be used as development completion criteria because DO may not stabilize.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry three times in succession and the turbidity has decreased, or per the requirements set forth in the project-specific SAP. Water quality parameters may be recorded if feasible using the flow-through cell.

If any water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development, or per the requirements set forth in the project-specific SAP.

7.4 Development of Wells with Low Yield

Water is the primary mechanism to remove fines and flush water through the gravel pack for effective development. Therefore, development can be a challenge in wells that do not yield sufficient water to recharge when water is removed. However, often these wells are the most in need of development to improve their performance as they are typically installed in low permeability formations with a high content of fines. Development of these wells can improve their yield.

The surging portion of the development can be successfully performed in a well with standing water regardless of its yield. It is the subsequent removal of fine materials that is hindered when insufficient

water is recharged to the well. When wells go dry or drawdown significantly during development, development can be performed intermittently, allowing sufficient water to recharge prior conducting the next stage of surging. These intermittent procedures can take place hours or even days apart, depending on project-specific time constraints.

7.5 Wells containing NAPL

Additional care should be taken when planning development of wells that contain NAPL. If the NAPL is flammable, there are health and safety as well as handling issues to consider. If NAPL in excess of a persistent sheen is noted, the recharge rate will be evaluated through hand bailing. In most cases, it is generally preferable to remove NAPL by bailing to the extent practical prior to performing development. Groundwater parameters, excluding turbidity, will not be collected during well development if NAPL or excessive sheen is noticed in the purged water during development to ensure the meter probes are not fouled or destroyed. Well development will be halted.

Development by surging or pumping the well dry can result in the spreading of NAPL vertically in the soil column around the well. These methods can be used, if information exists describing the vertical thickness of the NAPL smear zone around the well, and if the methods do not result in mounding or drawdown that exceeds this thickness. Alternate methods such as bailing may also be used, but any method should not allow the well to be pumped dry or result in significant drawdown that would spread the NAPL vertically.

7.6 Temporary Well Points

For certain projects, temporary well points (TWPs) may be installed to collect groundwater samples at a site. Since no sand pack, bentonite chips, or bentonite grout are generally used in the construction of the TWPs, development can proceed as soon as sufficient water has entered the well to static conditions. Due to the small diameter of these wells, generally ¾-inch to 1-inch ID, development will be performed using either a small diameter (0.5-inch) bailer and/or a peristaltic pump with dedicated tubing. The TWPs will have minimal water column and may purge dry during development. However, attempts will be made to remove fines from the well prior to sampling. Purging and sampling may occur as soon as approximately 80% of the static water has re-entered the TWP, or per the requirements set forth in the project-specific SAP.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP.

8.2 Quality control (QC) requirements are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for equipment decontamination (frequency and materials) and IDW handling.

9.0 Records, Data Analysis, Calculations

9.1 All data and information (e.g., development method used) must be documented on field data sheets (Attachment 1) or within site logbooks with permanent ink. Data recorded may include the following:

- Well Location
- Weather conditions
- Date and Time
- Purge Method
- Reading/measurements obtained

10.0 Attachments or References

Attachment 1 – Well Development Record

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

Attachment 1
Well Development Record

AECOM | Any Task • Anywhere | TIDEWATER INC

	PROJECT NUMBER	WELL NUMBER
		SHEET OF
	WELL PURGE AND SAMPLING FIELD SHEET	

	CASING DIAMETER	GAL/FT OF CASING
DEPTH TO WATER (FT): =		
	1 IN.	**0.0408**
WELL DEPTH (FT):	**2 IN.**	**0.1632**
WATER COLUMN (FT): =	**4 IN.**	**0.6528**
GAL/FT OF CASING x	**6 IN.**	**1.4688**
CASING VOLUME (GAL) =	**8 IN.**	**2.611**
NO. OF VOLUMES min.(3) x	**10 IN.**	**4.0797**
PURGE VOLUME (GAL) =	**12 IN.**	**5.8748**

METHOD OF PURGING (circle one)

PUMP: SUB. BLADDER. PERIST.	**OTHER:**	**BAILER** : TEFLON, SS ,OTHER:
TIME ON:		BAILER VOL.. (gal)
FLOW RATE (ml/min):		REQUIRED PULLS:
PUMP TIME (min):		VOL. PURGED (gals):
VOL. PURGED (gals):		OTHER:

Date Time	Water Volume Discharged (gal)	Water Level (ft BTOC)	Turbidity (NTU)	Temperature (°C)	pH	Conductivity (mS/cm)	ORP mV	Dissolved Oxygen mg/L	Remarks (color, odor, sheen, sediment, etc.)

MP-Purge Form

Monitoring Well Sampling

Procedure 3-14

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the actions to be used during monitoring well sampling activities and establishes the method for sampling groundwater monitoring wells for water-borne contaminants and general groundwater chemistry. The objective is to obtain groundwater samples that are representative of aquifer conditions with as little alteration to water chemistry as possible.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first well. All field sampling personnel responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the well sampling tasks. Conduct preliminary area monitoring of sampling wells to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and liquid matrix through the use of appropriate personal protective equipment (PPE).

2.2 Observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable nitrile or vinyl gloves, outer chemical-protective nitrile gloves and rubberized safety-toed boots. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations. Refer to the project-specific HASP for the required PPE.

2.3 Physical Hazards associated with Well Sampling

- To avoid lifting injuries associated with pump and bailers retrieval, use the large muscles of the legs, not the back.
- Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
- When using tools for cutting purposes, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid slip/trip/fall conditions as a result of pump discharge, use textured boots/boot cover bottoms.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- Be aware of restricted mobility due to PPE.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross-contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 **The Delivery Order (DO) Manager is responsible for ensuring that monitoring well sampling activities comply with this procedure. The DO Manager is responsible for ensuring that all field sampling personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks.**

5.2.2 **The Program Quality Manager is responsible for ensuring overall compliance with this procedure.**

5.2.3 **The Field Manager is responsible for ensuring that all field sampling personnel follow these procedures.**

5.2.4 **Field sampling personnel are responsible for the implementation of this procedure.**

5.2.5 The field sampler and/or task manager is responsible for directly supervising the groundwater sampling procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 Purging and Sampling Equipment

- Pump (Peristaltic, Portable Bladder, Submersible)
- Polyethylene or Teflon bladders (for portable bladder pumps)
- Bladder pump controller (for portable bladder pumps)
- Air compressor (for portable bladder pumps)
- Nitrogen cylinders (for portable bladder pumps)
- 12-volt power source
- Polyethylene inlet and discharge tubing (except for VOC analysis which requires Teflon tubing)
- Silicone tubing appropriate for peristaltic pump head
- Teflon bailer appropriately sized for well
- Disposable bailer string (polypropylene)
- Individual or multi-parameter water quality meter(s) with flow-through cell to measure temperature, pH, specific conductance, dissolved oxygen (DO), oxidation reduction potential (ORP), and/or turbidity
- Turbidity meter
- Water level meter
- Oil/water interface probe

6.2 General Equipment

- Sample kit (i.e., bottles, labels, preservatives, custody records and tape, cooler, ice)
- Sample Chain-of-Custody (COC) forms
- Sample Collection Records

- Sample packaging and shipping supplies
- Waterproof marker or paint
- Distilled/deionized water supply
- Water dispenser bottles
- Flow measurement cup or bucket
- 5-gallon buckets
- Instrument calibration solutions
- Stopwatch or watch
- Disposable Nitrile gloves
- Paper towels
- Trash bags
- Zipper-lock bags
- Equipment decontamination supplies
- Health and safety supplies (as required by the HASP)
- Approved plans such as: project-specific HASP and Sampling and Analysis Plan (SAP)
- Well keys or combinations
- Monitoring well location map(s)
- Field project logbook/pen

7.0 Calibration or Standardization

7.1 Field instruments will be calibrated daily according to the requirements of the SAP and manufacturer's specifications for each piece of equipment. Equipment will be checked daily with the calibration solutions at the end of use of the equipment. Calibration records shall be recorded in the field logbook or appropriate field form.

7.2 If readings are suspected to be inaccurate, the equipment shall be checked with the calibration solutions and/or re-calibrated.

8.0 Procedure

8.1 **Preparation**

8.1.1 **Site Background Information**

Establish a thorough understanding of the purposes of the sampling event prior to field activities. Conduct a review of all available data obtained from the site and pertinent to the water sampling. Review well history data including, but not limited to, well locations, sampling history, purging rates, turbidity problems, previously used purging methods, well installation methods, well completion records, well development methods, previous analytical results, presence of an immiscible phase, historical water levels, and general hydrogeologic conditions.

Previous groundwater development and sampling logs give a good indication of well purging rates and the types of problems that might be encountered during sampling, such as excessive turbidity and low well yield. They may also indicate where dedicated pumps are placed in the water column. To help minimize the potential for cross-contamination, well purging and sampling and water level measurement collection shall proceed from the least contaminated to the most contaminated well as indicated by previous analytical results. This order may be changed in the field if conditions warrant it, particularly if dedicated sampling equipment is used. A review of prior sampling procedures and results may also identify which purging and sampling techniques are appropriate for the parameters to be tested under a given set of field conditions.

8.1.2 Groundwater Analysis Selection

Establish the requisite field and laboratory analyses prior to water sampling. Decide on the types and numbers of quality assurance/quality control (QA/QC) samples to be collected (refer to the project-specific SAP), as well as the type and volume of sample preservatives, the type and number of sample containers, the number of coolers required, and the quantity of ice or other chilling materials. The field sampling personnel shall ensure that the appropriate number and size sample containers are brought to the site, including extras in case of breakage or unexpected field conditions. Refer to the project-specific SAP for the project analytical requirements.

8.2 Groundwater Sampling Procedures

Groundwater sampling procedures at a site shall include:

1) An evaluation of the well security and condition prior to sampling;
2) Decontamination of equipment;
3) Measurement of well depth to groundwater;
4) Assessment of the presence or absence of an immiscible phase;
5) Assessment of purge parameter stabilization;
6) Purging of static water within the well and well bore; and
7) Obtaining a groundwater sample.

Each step is discussed in sequence below. Depending upon specific field conditions, additional steps may be necessary. As a rule, at least 24 hours should separate well development and well sampling events. In all cases, consult the State and local regulations for the site, which may require more stringent time separation between well development and sampling.

8.2.1 Well Security and Condition

At each monitoring well location, observe the conditions of the well and surrounding area. The following information may be noted on a Groundwater Sample Collection Record (Attachment 1) or in the field logbook:

- Condition of the well's identification marker.
- Condition of the well lock and associated locking cap.
- Integrity of the well – well pad condition, protective outer casing, obstructions or kinks in the well casing, presence of water in the annular space, and the top of the interior casing.
- Condition of the general area surrounding the well.

8.2.2 Decontamination of Equipment

Where possible, dedicated supplies should be used at each well location to minimize the potential for cross-contamination and minimize the amount of investigation derived waste (IDW) fluids resulting from the decontamination process. If decontamination is necessary, establish a decontamination station before beginning sampling. The station shall consist of an area of at least 4 feet by 2 feet covered with plastic sheeting and be located upwind of the well being sampled. The station shall be large enough to fit the appropriate number of wash and rinse buckets, and have sufficient room to place equipment after decontamination. One central cleaning area may be used throughout the entire sampling event. The area around the well being sampled shall also be covered with plastic sheeting to prevent spillage. Further details are presented in SOP 3-06, Equipment Decontamination.

Decontaminate each piece of equipment prior to entering the well. Also, conduct decontamination prior to sampling at a site, even if the equipment has been decontaminated subsequent to its last usage. Additionally, decontaminate each piece of equipment used at the site prior to leaving the site. It is only necessary to decontaminate dedicated sampling equipment prior to installation within the well. Do not place clean sampling equipment directly on the ground or other contaminated surfaces prior to insertion into the well. Dedicated sampling equipment that has been certified by the manufacturer as being decontaminated can be placed in the well without on-site decontamination.

8.2.3 Measurement of Static Water Level Elevation

Before purging the well, measure water levels in all of the wells within the zone of influence of the well being purged. The best practice, if possible, is to measure all site wells (or wells within the monitoring well network) prior to sampling. If the well cap is not vented, remove the cap several minutes before measurement to allow water levels to equilibrate to atmospheric pressure.

Measure the depth to standing water and the total depth of the well to the nearest 0.01 foot to provide baseline hydrologic data, to calculate the volume of water in the well, and to provide information on the integrity of the well (e.g., identification of siltation problems). If not already present, mark an easily identified reference point for water level measurements which will become the measuring point for all water level measurements. This location and elevation must be surveyed.

The device used to measure the water level surface and depth of the well shall be sufficiently sensitive and accurate in order to obtain a measurement to the nearest 0.01 foot reliably. An electronic water level meter will usually be appropriate for this measurement; however, when the groundwater within a particular well is highly contaminated, an inexpensive weighted tape measure can be used to determine well depth to prevent adsorption of contaminants onto the meter tape. The presence of light, non-aqueous phase liquids (LNAPLs) and/or dense, non-aqueous phase liquids (DNAPLs) in a well requires measurement of the elevation of the top and the bottom of the product, generally using an interface probe. Water levels in such wells must then be corrected for density effects to accurately determine the elevation of the water table.

At each location, measure water levels several times in quick succession to ensure that the well has equilibrated to atmospheric conditions prior to recording the measurement. As stated above, measure all site wells (or wells within the monitoring well network) prior to sampling whenever possible. This will provide a water level database that describes water levels across the site at one time (a synoptic sampling). Prior to sampling, measure the water level in each well immediately prior to purging the well to ascertain that static conditions have been achieved prior to sampling.

8.2.4 Detection of Immiscible Phase Layers

Complete the following steps for detecting the presence of LNAPL and DNAPL before the well is purged for conventional sampling. These procedures may not be required for all wells. Consult the project-specific SAP to determine if assessing the presence of LNAPL and/or DNAPL is necessary.

1) Sample the headspace in the wellhead immediately after the well is opened for organic vapors using either a PID or an organic vapor analyzer, and record the measurements.

2) Lower an interface probe into the well to determine the existence of any immiscible layer(s), LNAPL and/or DNAPL, and record the measurements.

3) Confirm the presence or absence of an immiscible phase by slowly lowering a clear bailer to the appropriate depth, then visually observing the results after sample recovery.

4) In rare instances, such as when very viscous product is present, it may be necessary to utilize hydrocarbon- and water-sensitive pastes for measurement of LNAPL thickness. This is accomplished by smearing adjacent, thin layers of both hydrocarbon- and water-sensitive pastes along a steel measuring tape and inserting the tape into the well. An engineering tape showing tenths and hundredths of feet is required. Record depth to water, as shown by the mark on the water-sensitive paste, and depth to product, as shown by the mark on the product-sensitive paste. In wells where the approximate depth to water and product thickness are not known, it is best to apply both pastes to the tape over a fairly long interval (5 feet or more). Under these conditions, measurements are obtained by trial and error and may require several insertions and retrievals of the tape before the paste-covered interval of the tape encounters product and water. In wells where approximate depths of air-product and product-water interfaces are known, pastes may be applied over shorter intervals. Water depth measurements should not be used in preparation of water table contour maps until they are corrected for depression by the product.

5) If the well contains an immiscible phase, it may be desirable to sample this phase separately. Section 8.2.6 presents immiscible phase sampling procedures. It may not be meaningful to conduct water sample analysis of water obtained from a well containing LNAPLs or DNAPLs. Consult the **DO Manager** and **Program Quality Manager** if this situation is encountered.

8.2.5 **Purging Equipment and Use**

General Requirements

The water present in a well prior to sampling may not be representative of in situ groundwater quality and shall be removed prior to sampling. Handle all groundwater removed from potentially contaminated wells in accordance with the IDW handling procedures in SOP 3-05, IDW Management. Purging shall be accomplished by methods as indicated in the project-specific SAP or by those required by State requirements. For the purposes of this SOP, purging methods will be described by removing groundwater from the well using low-flow techniques.

According to the U.S. Environmental Protection Agency (EPA) (EPA, 1996), the rate at which groundwater is removed from the well during purging ideally should be less than 0.2 to 0.3 liters/minute. EPA further states that wells should be purged at rates below those used to develop the well to prevent further development of the well, to prevent damage to the well, and to avoid disturbing accumulated corrosion or reaction products in the well. EPA also indicates that wells should be purged at or below their recovery rate so that migration of water in the formation above the well screen does not occur.

Realistically, the purge rate should be low enough that substantial drawdown in the well does not occur during purging. In addition, a low purge rate will reduce the possibility of stripping volatile organic compounds (VOCs) from the water, and will reduce the likelihood of increasing the turbidity of the sample due to mobilizing colloids in the subsurface that are immobile under natural flow conditions.

The field sampler shall ensure that purging does not cause formation water to cascade down the sides of the well screen. Wells should not be purged to dryness if recharge causes the formation water to cascade down the sides of the screen, as this will cause an accelerated loss of volatiles. This problem should be anticipated based on the results of either the well development task or historical sampling events. In general, place the intake of the purge pump in the middle of the saturated screened interval within the well to allow purging and at the same time minimize disturbance/overdevelopment of the screened interval in the well. Water shall be purged from the well at a rate that does not cause recharge water to be excessively agitated unless an extremely slow recharging well is encountered where complete evacuation is unavoidable. During the well purging procedure, collect water level and/or product level measurements to assess the hydraulic effects of purging. Sample the well when it recovers sufficiently to provide enough water for the analytical parameters specified. If the well is purged dry, allow the well to recover sufficiently to provide enough water for the specified analytical parameters, and then sample it.

Evaluate water samples on a regular basis during well purging and analyze them in the field preferably using in-line devices (i.e., flow through cell) for temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation-reduction (redox) potential. Turbidity may be measured using in-line devise with a built in turbidity probe or separately (outside of the flow-through cell) with a nephelometer or similar device.

Readings should be taken every 2 to 5 minutes during the purging process. These parameters are measured to demonstrate that the natural character of the formation waters has been restored.

Purging shall be considered complete per the requirements set forth in the project-specific SAP, State requirements, or when three consecutive field parameter measurements of temperature, pH, specific conductivity, DO and ORP stabilize within approximately 10 percent and the turbidity is at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU. This criterion may not be applicable to temperature if a submersible pump is used during purging due to the heating of the water by the pump motor. Enter all information obtained during the purging and sampling process into a groundwater sampling log. Attachment 1 shows an example of a groundwater sampling log and the information typically included in the form. Whatever form is used, all blanks need to be completed on the field log during field sampling.

Groundwater removed during purging shall be stored according to the project-specific SAP or per SOP 3-05, IDW Management.

Purging Equipment and Methods

Submersible Pump

A stainless steel submersible pump may be utilized for purging both shallow and deep wells prior to sampling the groundwater for semivolatile and non-volatile constituents, but are generally not preferred for VOCs unless there are no other options (e.g., well over 200 feet deep). For wells over 200 feet deep,

the submersible pump is one of the few technologies available to feasibly accomplish purging under any yield conditions. For shallow wells with low yields, submersible pumps are generally inappropriate due to overpumpage of the wells (<1 gallon per minute), which causes increased aeration of the water within the well.

Steam clean or otherwise decontaminate the pump and discharge tubing prior to placing the pump in the well. The submersible pump shall be equipped with an anti-backflow check valve to limit the amount of water that will flow back down the drop pipe into the well. Place the pump in the middle of the saturated screened interval within the well and maintain it in that position during purging.

Bladder Pump

A stainless steel bladder pump can be utilized for purging and sampling wells up to 200 feet in depth for volatile, semivolatile, and non-volatile constituents. Use of the bladder pump is most effective in low to moderate yield wells and are often the preferred method for low-flow sampling. When sampling for VOCs and/or SVOCs, Teflon bladders should be used. Polyethylene bladders may be used when sampling for inorganics.

Either compressed dry nitrogen or compressed dry air, depending upon availability, can operate the bladder pump. The driving gas utilized must be dry to avoid damage to the bladder pump control box. Decontaminate the bladder pump prior to use.

Centrifugal, Peristaltic, or Diaphragm Pump

A centrifugal, peristaltic, or diaphragm pump may be utilized to purge a well if the water level is within 27 feet of ground surface. New or dedicated tubing is inserted into the midpoint of the saturated screened interval of the well. Water should be purged at a rate that satisfies low-flow requirements (i.e., does not cause drawdown). Centrifugal, peristaltic, or diaphragm pump are generally discouraged for VOCs sampling; however, follow methods allowed per the project-specific SAP or State requirements.

Air Lift Pump

Airlift pumps are not appropriate for purging or sampling.

Bailer

Avoid using a bailer to purge a well because it can result in overdevelopment of the well and create excessive purge rates. If a bailer must be used, the bailer should either be dedicated or disposable. Teflon-coated cable mounted on a reel is recommended for lowering the bailer in and out of the well.

Lower the bailer below the water level of the well with as little disturbance of the water as possible to minimize aeration of the water in the well. One way to gauge the depth of water on the reel is to mark the depth to water on the bailer wire with a stainless steel clip. In this manner, less time is spent trying to identify the water level in the well.

8.2.6 **Monitoring Well Sampling Methodologies**

<u>Sampling Light, Non-Aqueous Phase Liquids (LNAPL)</u>

Collect LNAPL, if present, prior to any purging activities. The sampling device shall generally consist of a dedicated or disposable bailer equipped with a bottom-discharging device. Lower the bailer slowly until contact is made with the surface of the LNAPL, and to a depth less than that of the immiscible fluid/water interface depth as determined by measurement with the interface probe. Allow the bailer to fill with LNAPL and retrieve it.

When sampling LNAPLs, never drop bailers into a well and always remove them from the well in a manner that causes as little agitation of the sample as possible. For example, the bailer should not be removed in a jerky fashion or be allowed to continually bang against the well casing as it is raised. Teflon bailers should always be used when sampling LNAPL. The cable used to raise and lower the bailer shall be composed of an inert material (e.g., stainless steel) or coated with an inert material (e.g., Teflon).

<u>Sampling Dense, Non-Aqueous Phase Liquids (DNAPL)</u>

Collect DNAPL prior to any purging activities. The best method for collecting DNAPL is to use a double-check valve, stainless steel bailer, or a Kemmerer (discrete interval) sampler. The sample shall be collected by slow, controlled lowering of the bailer to the bottom of the well, activation of the closing device, and retrieval.

Groundwater Sampling Methodology

The well shall be sampled when groundwater within it is representative of aquifer conditions per the methods described in Section 8.2.5. Prior to sampling the flow-through cell shall be removed and the samples collected directly from the purge tubing. Flow rates shall not be adjusted once aquifer conditions are met. Additionally, a period of no more than 2 hours shall elapse between purging and sampling to prevent groundwater interaction with the casing and atmosphere. This may not be possible with a slowly recharging well. Measure and record the water level prior to sampling in order to monitor drawdown when using low-flow techniques and gauge well volumes removed and recharged when using non-low-flow techniques.

Sampling equipment (e.g., especially bailers) shall never be dropped into the well, as this could cause aeration of the water upon impact. Additionally, the sampling methodology utilized shall allow for the collection of a groundwater sample in as undisturbed a condition as possible, minimizing the potential for volatilization or aeration. This includes minimizing agitation and aeration during transfer to sample containers, minimizing exposure to sunlight, and immediately placing the sample on ice once collected.

Sampling equipment shall be constructed of inert material. Equipment with neoprene fittings, polyvinyl chloride (PVC) bailers, Tygon® tubing, silicon rubber bladders, neoprene impellers, polyethylene, and Viton® are not acceptable when sampling for organics. If bailers are used, an inert cable/chain (e.g., fluorocarbon resin-coated wire or stainless steel wire or cable) shall be used to raise and lower the bailer. Dedicated equipment is highly recommended for all sampling programs.

Submersible Pumps

The submersible pump must be specifically designed for groundwater sampling (i.e., pump composed of stainless steel and Teflon, sample discharge lines composed of Teflon) and must have a controller mechanism allowing the required low-flow rate. Adjust the pump rate so that flow is continuous and does not pulsate to avoid aeration and agitation within the sample discharge lines. Run the pump for several minutes at the low-flow rate used for sampling to ensure that the groundwater in the lines was obtained at the low-flow rate.

Bladder Pumps

A gas-operated stainless steel bladder pump with adjustable flow control and equipped with a Teflon bladder and Teflon-lined tubing can be effectively utilized to collect a groundwater sample and is considered to be the best overall device for sampling inorganic and organic constituents. If only inorganics are being sampled, polyvinyl bladders and tubing may be used. Operate positive gas displacement bladder pumps in a continuous manner so that they minimize discharge pulsation that can aerate samples in the return tube or upon discharge.

When using a compressor, take several precautions. If the compressor is being powered by a gasoline generator, position the generator downwind of the well. Ground fault circuit interrupters (GFCIs) should always be used when using electric powered equipment. Do not connect the compression hose from the compressor to the pump controller until after the engine has been started.

When all precautions are completed and the compressor has been started, connect the compression hose to the pump controller. Slowly adjust the control knobs to discharge water in the shortest amount of time while maintaining a near constant flow. This does not mean that the compressor must be set to discharge the water as hard as possible. The optimal setting is one that produces the largest volume of purge water per minute (not per purge cycle) while maintaining a near constant flow rate.

Prior to sampling, adjust the flow rate (purge rate) to yield 100 to 300 mL/minute. Avoid settings that produce pulsating streams of water instead of a steady stream if possible. Operate the pump at this low flow rate for several minutes to ensure that drawdown is not occurring. At no time shall the sample flow rate exceed the flow rate used while purging.

For those samples requiring filtration, it is recommended to use an in-line high capacity filter after all non-filtered samples have been collected.

***Peristaltic Pumps*:**

A peristaltic pump is a type of positive displacement pump that moves water via the process of peristalsis. The pump uses a flexible hose fitted inside a circular pump casing. A rotor with cams compresses the flexible tube as the rotor turns, which forces the water to be pumped to move through the tube. In peristaltic pumps, no moving parts of the pump are in contact with the water being pumped.

Displacement is determined by tube size, so delivery rate can only be changed during operation by varying pump speed. Peristaltic pumps are simple and quite inexpensive for the flow rates they provide.

There are several methods available for transferring the sample into the laboratory containers. The selected method may vary based on State requirements and should be documented in the project-specific SAP. Samples typically can be collected directly from the discharge end of the Teflon tubing, after it has been disconnected from the flow through cell. For volatile analyses, the sampler should make sure that the pump is set such that a smooth laminar flow is achieved. In all cases, the project team should consult their local regulatory requirements and document the selected sample collection procedure in the project-specific SAP.

Bailers

A single- or double-check valve Teflon or stainless steel bailer equipped with a bottom discharging device can be utilized to collect groundwater samples. Bailers have a number of disadvantages, however, including a tendency to alter the chemistry of groundwater samples due to degassing, volatilization, and aeration; the possibility of creating high groundwater entrance velocities; differences in operator techniques resulting in variable samples; and difficulty in determining where in the water column the sample was collected. Therefore, use bailers for groundwater sampling only when other types of sampling devices cannot be utilized for technical, regulatory, or logistical reasons.

Dedicated or disposable bailers should always be used in order to eliminate the need for decontamination and to limit the potential of cross-contamination. Each time the bailer is lowered to the water table, lower it in such a way as to minimize disturbance and aeration of the water column within the well.

8.2.7 Sample Handling and Preservation

Many of the chemical constituents and physiochemical parameters to be measured or evaluated during groundwater monitoring programs are chemically unstable and require preservation. The U.S. EPA document entitled, *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods (SW-846)* (EPA 1997), includes a discussion of appropriate sample preservation procedures. In addition, SW-846 provides guidance on the types of sample containers to use for each constituent or common set of parameters. In general, check with specific laboratory or State requirements prior to obtaining field samples. In many cases, the laboratory will supply the necessary sample bottles and required preservatives. In some cases, the field sampling personnel may add preservatives in the field.

Improper sample handling may alter the analytical results of the sample. Therefore, transfer samples in the field from the sampling equipment directly into the container that has been prepared specifically for that analysis or set of compatible parameters as described in the project-specific SAP. It is not an acceptable practice for samples to be composited in a common container in the field and then split in the laboratory, or poured first into a wide mouth container and then transferred into smaller containers.

Collect groundwater samples and place them in their proper containers in the order of decreasing volatility and increasing stability. A preferred collection order for some common groundwater parameters is:

1. VOCs and total organic halogens (TOX)
2. Dissolved gases, total organic carbon (TOC), total fuel hydrocarbons
3. Semivolatile organics, pesticides
4. Total metals, general minerals (unfiltered)
5. Dissolved metals, general minerals (filtered)
6. Phenols
7. Cyanide
8. Sulfate and chloride
9. Nitrate and ammonia
10. Radionuclides

When sampling for VOCs, collect water samples in vials or containers specifically designed to prevent loss of VOCs from the sample. The analytical laboratory performing the analysis shall provide these vials.

Collect groundwater from the sampling device in vials by allowing the groundwater to slowly flow along the sides of the vial. Sampling equipment shall not touch the interior of the vial. Fill the vial above the top of the vial to form a positive meniscus with no overflow. No headspace shall be present in the sample container once the container has been capped. This can be checked by inverting the bottle once the sample is collected and tapping the side of the vial to dislodge air bubbles. Sometimes it is not possible to collect a sample without air bubbles, particularly water that has high concentrations of dissolved gasses. In these cases, the field sampling personnel shall document the occurrence in the field logbook and/or sampling worksheet at the time the sample was collected. Likewise, the analytical laboratory shall note in the laboratory analysis reports any headspace in the sample container(s) at the time of receipt by the laboratory.

Special Handling Considerations

In general, samples for organic analyses should not be filtered. However, high turbidity samples for PCB analysis may require filtering. Consult the project-specific SAP for details on filtering requirements. Samples shall not be transferred from one container to another because this could cause aeration or a loss of organic material onto the walls of the container. TOX and TOC samples should be handled in the same manner as VOC samples.

When collecting total and dissolved metals samples, the samples should be collected sequentially. The total metals sample is collected from the pump unfiltered. The dissolved metals sample is collected after filtering with a 0.45-micron membrane in-line filter. Allow at least 500 mL of effluent to flow through the filter prior to sampling to ensure that the filter is thoroughly wetted and seated in the filter capsule. If required by the project-specific SAP, include a filter blank for each lot of filters used and always record the lot number of the filters.

Field Sampling Preservation

Preserve samples immediately upon collection. Ideally, sampling containers will be pre-preserved with a known concentration and volume of preservative. Certain matrices that have alkaline pH (greater than 7) may require more preservative than is typically required. An early assessment of preservation techniques, such as the use of pH strips after initial preservation, may therefore be appropriate. Guidance for the preservation of environmental samples can be found in the U.S. EPA *Handbook for Sampling and Sample Preservation of Water and Wastewater* (EPA 1982). Additional guidance can be found in other U.S. EPA documents (EPA 1992, 1996).

Field Sampling Log

A groundwater sampling log provided as Attachment 1 shall document the following:

- Identification of well
- Well depth
- Static water level depth and measurement technique
- Presence of immiscible layers and detection method
- Well yield
- Purge volume and pumping rate
- Time that the well was purged
- Sample identification numbers
- Well evacuation procedure/equipment
- Sample withdrawal procedure/equipment
- Date and time of collection
- Types of sample containers used
- Preservative(s) used
- Parameters requested for analysis
- Field analysis data

- Field observations on sampling event
- Name of sampler
- Weather conditions

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

10.0 Data and records management

10.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Sample Collection Records;
- Field logbook;
- Chain-of-custody forms; and
- Shipping labels.

10.2 Sample collection records (Attachment 1) will provide descriptive information for the purging process and the samples collected at each monitoring well.

10.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.

10.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

10.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

11.0 Attachments or References

Attachment 1 – Groundwater Sampling Collection Record

ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

Environmental Protection Agency, United States (EPA). 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater.* EPA-600/4-82-029. Cincinnati: EPA Office of Research and Development, Environmental Monitoring and Support Laboratory.

EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

EPA. 1996. *Ground Water Issue: Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. EPA/540/S-95/504. Office of Solid Waste and Emergency Response. April.

EPA. 1997. *Test Methods for Evaluating Solid Waste, Physical/Chemical Method (SW-846)*. 3rd ed., Final Update IIIA. Office of Solid Waste. Online updates at: http://www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

DoD Environmental Field Sampling Handbook, Revision 1.0. April 2013

SOP 3-03, *Recordkeeping, Sample Labelling, and Chain-of-Custody*.

SOP 3-05, *IDW Management.*

SOP 3-06, *Equipment Decontamination*.

Attachment 1
Groundwater Sample Collection Record

GROUNDWATER SAMPLE COLLECTION FIELD SHEET

GENERAL INFORMATION

SITE NAME _____ PROJECT NO. _____

SAMPLE NO. _____ WELL NO. _____

DATE/TIME COLLECTED _____ PERSONNEL _____

SAMPLE METHOD _____

SAMPLE MEDIA:

SAMPLE QC DUPLICATE: YES NO DUPLICATE SAMPLE NO. _____

MS/MSD REQUESTED YES NO MS/MSD SAMPLE NO.

SAMPLE CONTAINERS, PRESERVATIVES, ANALYSIS

Sample Container | Preservative | Analysis Requested

Volume per linear ft of casing		
ID (in)	Gallons	Liters
1	0.0408	0.1544
1 1/2	0.0918	0.3475
2	0.1632	0.6178
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600
8	2.611	9.8837

WELL PURGING DATA

Date _____
Time Started _____
Time Completed _____
PID Measurements
Background _____
Breathing Zone _____
Well Head _____
Purge Water

Well Depth (ft BTOC) _____
Depth to Water (ft BTOC) _____
Water Column Length _____
Well Casing Volume (per ft) _____
Volume of Water in Well (L) _____
Casing Volumes to Purge _____
Minimum to Purge (L) _____
Actual Purge (L) _____

FIELD MEASUREMENTS

Time	Amount Purged (gal)	pH	Temperature (Celsius)	Conductivity (mS/cm)	Dissolved Oxygen (mg/L)	ORP (mV)	Turbidity (NTU)	Depth to Water (ft BTOC)	Purge Rate (mL/min)

FIELD EQUIPMENT AND CALIBRATION

Equipment | Model | Calibration

GENERAL COMMENTS

Multi-Parameter Probe Unit # _____
Field Parameters Measured in Flow-Through Cell
Pump Placement Depth =
Pump Rate =
Well Diameter =
Screen Interval =

Soil and Rock Classification

Procedure 3-16

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) to thoroughly describe the physical characteristics of the sample and classify it according to the Unified Soil Classification System (USCS).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from AECOM-Tidewater JV, state and/or federal that are not addressed in this SOP and are applicable to soil and rock classification then those procedures may be added as an appendix to the project specific SAP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable nitrile or vinyl gloves, outer chemical-protective nitrile gloves, rubberized safety-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.

2.4 The health and safety considerations for the work associated with soil classification include:

- At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils/rocks without the use of gloves.
- Stay clear of all moving equipment and be aware of pinch points on machinery. Avoid wearing loose fitting clothing.
- When using cutting tools, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.

- To avoid heat/cold stress as a results of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and in case of extreme cold, wear insulating clothing.

3.0 Terms and Definitions

None.

4.0 Interference

None.

5.0 Training and Qualifications

5.1 The **Delivery Order (DO) Manager** is responsible for ensuring that the soil and rock classification procedures comply with this procedure. The **DO Manager** is responsible for ensuring that all personnel involved in soil and rock classification shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.3 The **Field Manager** is responsible for ensuring that all project **field personnel** follow these procedures.

5.4 Field personnel are responsible for the implementation of this procedure. Minimum qualifications for **field sampling personnel** require that one individual on the field team shall have a minimum of 6 months of experience with soil and rock classification.

5.5 The **project geologist** and/or **task manager** is responsible for directly supervising the soil and rock classification procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the **Program Quality Manager** and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials which may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the HASP
- Field log book and pen with indelible ink
- Boring log
- Munsell Soil Color Chart
- Scoopula, spatula, and/or other small hand tools
- California Sampler
- Hand-held penetrometer

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Soil Classification

The basic purpose of the classification of soil is to thoroughly describe the physical characteristics of the sample and to classify it according to an appropriate soil classification system. The USCS was developed so that soils could be described on a common basis by different investigators and serve as a

"shorthand" description of soil. A classification of a soil in accordance with the USCS includes not only a group symbol and name, but also a complete word description.

Describing soil on a common basis is essential so that soil described by different site qualified personnel is comparable. Site individuals describing soil as part of site activities *must* use the classification system described herein to provide the most useful geologic database for all present and future subsurface investigations and remedial activities.

The site geologist or other qualified individual shall describe the soil and record the description in a boring log, logbook, and/or electronic field data collection device. The essential items in any written soil description are as follows:

- Classification group name (e.g., silty sand)
- Color, moisture, and odor
- Range of particle sizes and maximum particle size
- Approximate percentage of boulders, cobbles, gravel, sand, and fines
- Plasticity characteristics of the fines
- In-place conditions, such as consistency, density, and structure
- USCS classification symbol

The USCS serves as ”shorthand“ for classifying soil into 15 basic groups:

- GW[1] Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines)
- GP[1] Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines)
- GM[1] Silty gravel (>50 percent gravel, >15 percent silt)
- GC[1] Clayey gravel (>50 percent gravel, >15 percent clay)
- SW[1] Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines)
- SP[1] Poorly graded (well sorted) sand (>50 percent sand, <5 percent fines)
- SM[1] Silty sand (>50 percent sand, >15 percent silt)
- SC[1] Clayey sand (>50 percent sand, >15 percent clay)
- ML[2] Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity)
- CL[2] Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity)
- MH[2] Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity)
- CH[2] Inorganic, high plasticity (fat) clay (no dilatancy, high toughness, and plasticity)
- OL Organic low plasticity silt or organic silty clay
- OH Organic high plasticity clay or silt
- PT Peat and other highly organic soil

Figure8-1 defines the terminology of the USCS. Flow charts presented in Figure 8-2 and indicate the process for describing soil. The particle size distribution and the plasticity of the fines are the two

[1] If percentage of fine is 5 percent to 15 percent, a dual identification shall be given (e.g., a soil with more than 50 percent poorly sorted gravel and 10 percent clay is designated GW-GC.

[2] If the soil is estimated to have 15 percent to 25 percent sand or gravel, or both, the words “with sand” or “with gravel” (whichever predominates) shall be added to the group name (e.g., clay with sand, CL; or silt with gravel, ML). If the soil is estimated to have 30 percent or more sand or gravel, or both, the words “sandy” or “gravely” (whichever predominates) shall be added to the group name (e.g., sandy clay, CL). If the percentage of sand is equal to the percent gravel, use “sandy.”

properties of soil used for classification. In some cases, it may be appropriate to use a borderline classification (e.g., SC/CL) if the soil has been identified as having properties that do not distinctly place the soil into one group.

8.1.1 Estimation of Particle Size Distribution

One of the most important factors in classifying a soil is the estimated percentage of soil constituents in each particle size range. Being proficient in estimating this factor requires extensive practice and frequent checking. The steps involved in determining particle size distribution are listed below:

1. Select a representative sample (approximately 1/2 of a 6-inch long by 2.5-inch diameter sample liner).
2. Remove all particles larger than 3 inches from the sample. Estimate and record the percent by volume of these particles. Only the fraction of the sample smaller than 3 inches is classified.
3. Estimate and record the percentage of dry mass of gravel (less than 3 inches and greater than 1/4 inch).
4. Considering the rest of the sample, estimate, and record the percentage of dry mass of sand particles (about the smallest particle visible to the unaided eye).
5. Estimate and record the percentage of dry mass of fines in the sample (do not attempt to separate silts from clays).
6. Estimate percentages to the nearest 5 percent. If one of the components is present in a quantity considered less than 5 percent, indicate its presence by the term “trace”.
7. The percentages of gravel, sand, and fines must add up to 100 percent. “Trace” is not included in the 100 percent total.

8.1.2 Soil Dilatancy, Toughness, and Plasticity

8.1.2.1 Dilatancy

To evaluate dilatancy, follow these procedures:

1. From the specimen, select enough material to mold into a ball about 1/2 inch (12 millimeters [mm]) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
2. Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 8-1. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

Table 8-1: Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

8.1.2.2 Toughness

Following the completion of the dilatancy test, shape the test specimen into an elongated pat and roll it by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. (If the sample is too wet to roll easily, spread it into a thin layer and allow it to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble at a diameter of 1/8 inch when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, lump the pieces together and knead it until the lump crumbles. Note the toughness of the

material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 8-2.

Table 8-2: Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread near the plastic limit. The thread and the lump have very high stiffness.

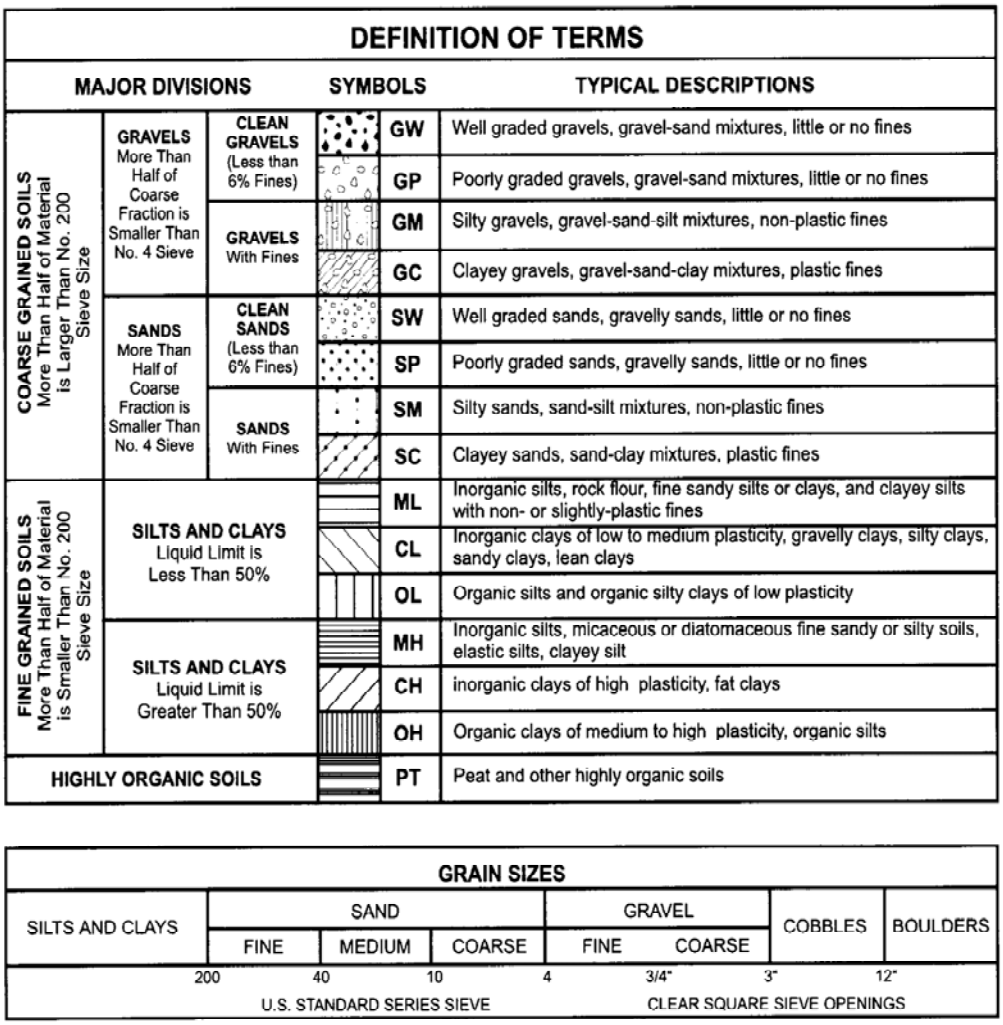


Figure8-1: Unclassified Soil Classification System (USCS)

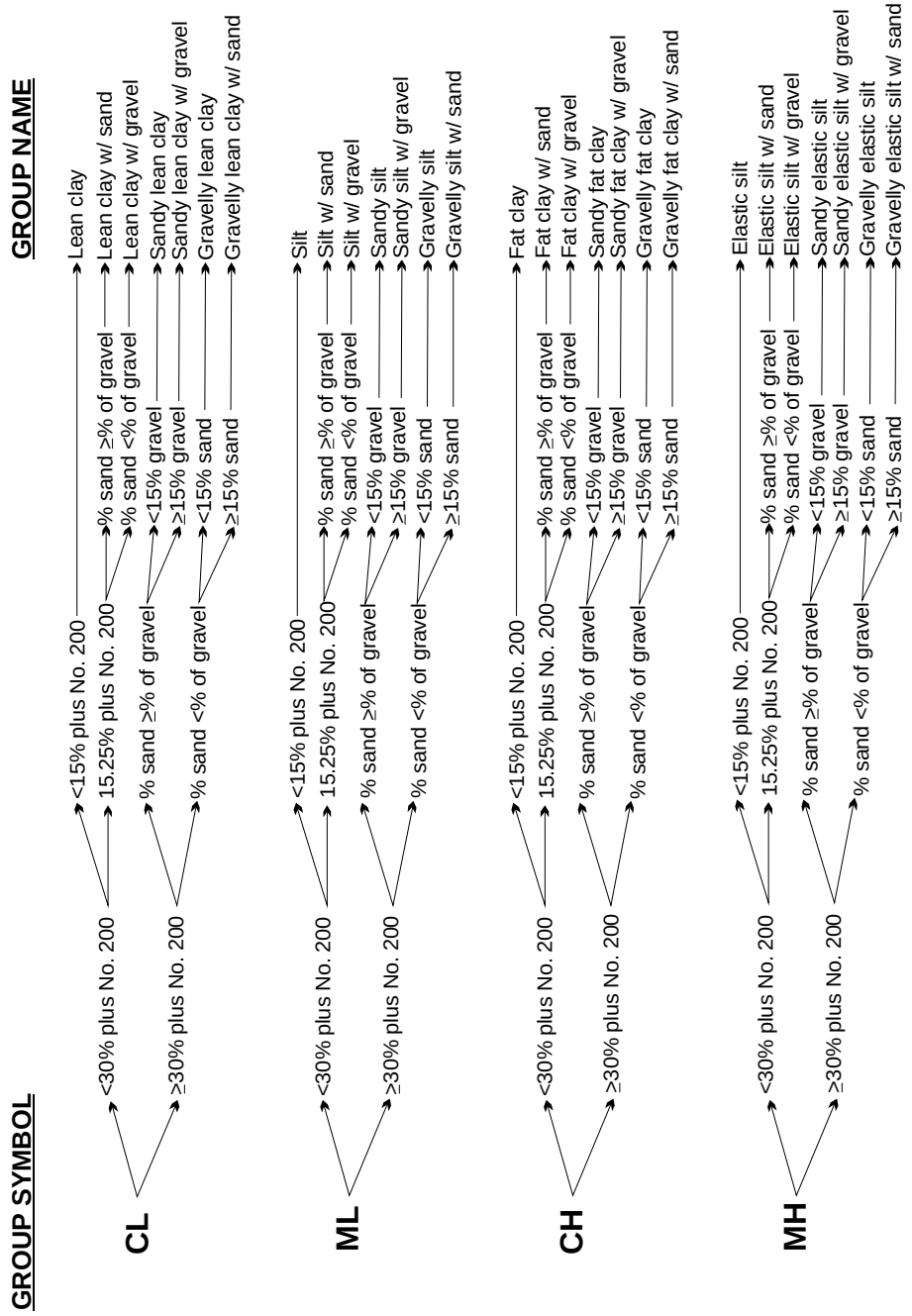


Figure 8-2: Flow Chart for Fine Grain Soil Classification

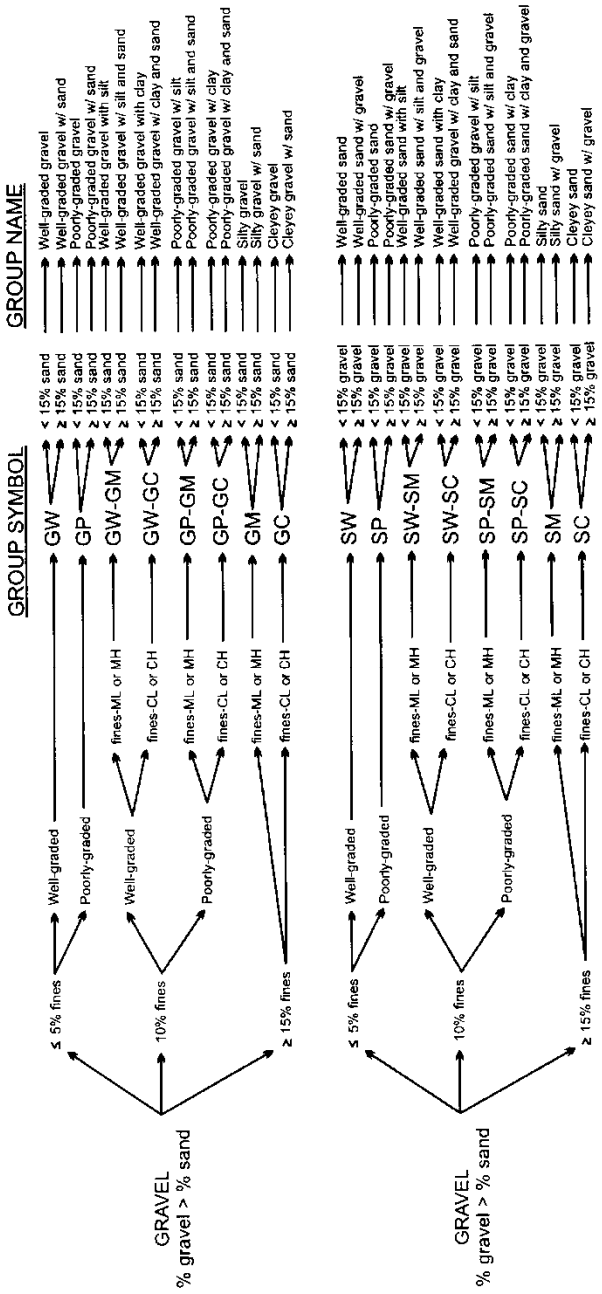


Figure 8-3: Flow Chart for Soil with Gravel

Plasticity

The plasticity of a soil is defined by the ability of the soil to deform without cracking, the range of moisture content over which the soil remains in a plastic state, and the degree of cohesiveness at the plastic limit. The plasticity characteristic of clays and other cohesive materials is defined by the liquid limit and plastic limit. The liquid limit is defined as the soil moisture content at which soil passes from the liquid to the plastic state as moisture is removed. The test for the liquid limit is a laboratory, not a field, analysis.

The plastic limit is the soil moisture content at which a soil passes from the plastic to the semi-solid state as moisture is removed. The plastic limit test can be performed in the field and is indicated by the ability to roll a 1/8-inch (0.125-inch) diameter thread of fines, the time required to roll the thread, and the number of times the thread can be re-rolled when approaching the plastic limit.

The plasticity tests are not based on natural soil moisture content, but on soil that has been thoroughly mixed with water. If a soil sample is too dry in the field, add water prior to performing classification. If a soil sample is too sticky, spread the sample thin and allow it to lose some soil moisture.

Table 8-3 presents the criteria for describing plasticity in the field using the rolled thread method.

Table 8-3: Criteria for Describing Plasticity

Description	Criteria
Non-Plastic	A 1/8-inch thread cannot be rolled.
Low Plasticity	The thread can barely be rolled.
Medium Plasticity	The thread is easy to roll and not much time is required to reach the plastic limit.
High Plasticity	It takes considerable time rolling the thread to reach the plastic limit.

8.1.3 Angularity

The following criteria describe the angularity of the coarse sand and gravel particles:

- **Rounded** particles have smoothly-curved sides and no edges.
- **Subrounded** particles have nearly plane sides, but have well-rounded corners and edges.
- **Subangular** particles are similar to angular, but have somewhat rounded or smooth edges.
- **Angular** particles have sharp edges and relatively plane sides with unpolished surfaces. Freshly broken or crushed rock would be described as angular.

8.1.4 Color, Moisture, and Odor

The natural moisture content of soil is very important. Table **8-4** shows the terms for describing the moisture condition and the criteria for each.

Table 8-4: Soil Moisture Content Qualifiers

Qualifier	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp but no visible water
Wet	Visible water, usually soil is below water table

Color is described by hue and chroma using the Munsell Soil Color Chart (Munsell 2000). For uniformity, all site geologists shall utilize this chart for soil classification. Doing so will facilitate correlation of geologic units between boreholes logged by different geologists. The Munsell Color Chart is a small booklet of numbered color chips with names like “5YR 5/6, yellowish-red.” Note mottling or banding of colors. It is particularly important to note and describe staining because it may indicate contamination.

In general, wear a respirator if strong organic odors are present. If odors are noted, describe them if they are unusual or suspected to result from contamination. An organic odor may have the distinctive smell of decaying vegetation. Unusual odors may be related to hydrocarbons, solvents, or other chemicals in the subsurface. An organic vapor analyzer may be used to detect the presence of volatile organic contaminants.

8.1.5 In-Place Conditions

Describe the conditions of undisturbed soil samples in terms of their density/consistency (i.e., compactness), cementation, and structure utilizing the following guidelines:

8.1.5.1 Density/Consistency

Density and consistency describe a physical property that reflects the relative resistance of a soil to penetration. The term “density” is commonly applied to coarse to medium-grained sediments (i.e., gravels,

sands), whereas the term “consistency” is normally applied to fine-grained sediments (i.e., silts, clays). There are separate standards of measure for both density and consistency that are used to describe the properties of a soil.

The density or consistency of a soil is determined by observing the number of blows required to drive a 1 3/8-inch (35 mm) diameter split barrel sampler 18 inches using a drive hammer weighing 140 lbs (63.5 kilograms [kg]) dropped over a distance of 30 inches (0.76 meters). Record the number of blows required to penetrate each 6 inches of soil in the field boring log during sampling. The first 6 inches of penetration is considered to be a seating drive; therefore, the blow count associated with this seating drive is recorded, but not used in determining the soil density/consistency. The sum of the number of blows required for the second and third 6 inches of penetration is termed the “standard penetration resistance,” or the “N-value.” The observed number of blow counts must be corrected by an appropriate factor if a different type of sampling device (e.g., Modified California Sampler with liners) is used. For a 2 3/8-inch inner diameter (I.D.) Modified California Sampler equipped with brass or stainless steel liners and penetrating a cohesionless soil (sand/gravel), the N-value from the Modified California Sampler must be divided by 1.43 to provide data that can be compared to the 1 3/8-inch diameter sampler data.

For a cohesive soil (silt/clay), the N-value for the Modified California Sampler should be divided by a factor of 1.13 for comparison with 1 3/8-inch diameter sampler data.

Drive the sampler and record blow counts for each 6-inch increment of penetration until one of the following occurs:

- A total of 50 blows have been applied during any one of the three 6-inch increments; a 50-blow count occurrence shall be termed “refusal” and noted as such on the boring log.
- A total of 150 blows have been applied.
- The sampler is advanced the complete 18 inches without the limiting blow counts occurring, as described above.

If the sampler is driven less than 18 inches, record the number of blows per partial increment on the boring log. If refusal occurs during the first 6 inches of penetration, the number of blows will represent the N-value for this sampling interval. Table 8-5 and Table 8-6 present representative descriptions of soil density/consistency vs. N-values.

Table 8-5: Measuring Soil Density with a California Sampler – Relative Density (Sands, Gravels)

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.43 factor
Very Loose	0–4	0–6
Loose	4–10	6–14
Medium Dense	10–30	14–43
Dense	30–50	43–71
Very Dense	> 50	> 71

Table 8-6: Measuring Soil Density with a California Sampler – Fine Grained Cohesive Soil

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.13 factor
Very Soft	0–2	0–2
Soft	2–4	2–4
Medium Stiff	4–8	4–9
Stiff	8–16	9–18
Very Stiff	16–32	18–36
Hard	> 32	> 36

For undisturbed fine-grained soil samples, it is also possible to measure consistency with a hand-held penetrometer. The measurement is made by placing the tip of the penetrometer against the surface of the soil contained within the sampling liner or shelby tube, pushing the penetrometer into the soil a distance specified by the penetrometer manufacturer, and recording the pressure resistance reading in pounds per square foot (psf). The values are as follows (Table 8-7):

Table 8-7: Measuring Soil Consistency with a Hand-Held Penetrometer

Description	Pocket Penetrometer Reading (psf)
Very Soft	0–250
Soft	250–500
Medium Stiff	500–1000
Stiff	1000–2000
Very Stiff	2000–4000
Hard	>4000

Consistency can also be estimated using thumb pressure using Table 8-8.

Table 8-8: Measuring Soil Consistency Using Thumb Pressure

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will penetrate soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

8.1.5.2 Cementation

Cementation is used to describe the friability of a soil. Cements are chemical precipitates that provide important information as to conditions that prevailed at the time of deposition, or conversely, diagenetic effects that occurred following deposition. Seven types of chemical cements are recognized by Folk (1980). They are as follows:

- Quartz – siliceous
- Chert – chert-cemented or chalcedonic
- Opal – opaline
- Carbonate – calcitic, dolomitic, sideritic (if in doubt, calcareous should be used)
- Iron oxides – hematitic, limonitic (if in doubt, ferruginous should be used)
- Clay minerals – if the clay minerals are detrital or have formed by recrystallization of a previous clay matrix, they are not considered to be a cement. Only if they are chemical precipitates, filling previous pore space (usually in the form of accordion-like stacks or fringing radial crusts) should they be included as “kaolin-cemented,” “chlorite-cemented,” etc.
- Miscellaneous minerals – pyritic, collophane-cemented, glauconite-cemented, gypsiferous, anhydrite-cemented, baritic, feldspar-cemented, etc.

The degree of cementation of a soil is determined qualitatively by utilizing finger pressure on the soil in one of the sample liners to disrupt the gross soil fabric. The three cementation descriptors are as follows:

- Weak – friable; crumbles or breaks with handling or slight finger pressure
- Moderate – friable; crumbles or breaks with considerable finger pressure
- Strong – not friable; will not crumble or break with finger pressure

8.1.5.3 Structure

This variable is used to qualitatively describe physical characteristics of soil that are important to incorporate into hydrogeological and/or geotechnical descriptions of soil at a site. Appropriate soil structure descriptors are as follows:

- Granular – spherically shaped aggregates with faces that do not accommodate adjoining faces
- Stratified – alternating layers of varying material or color with layers at least 6 mm (1/4 inch) thick; note thickness

- Laminated – alternating layers of varying material or color with layers less than 6 mm (1/4 inch) thick; note thickness
- Blocky – cohesive soil that can be broken down into small angular or subangular lumps that resist further breakdown
- Lensed – inclusion of a small pocket of different soil, such as small lenses of sand, should be described as homogeneous if it is not stratified, laminated, fissured, or blocky. If lenses of different soil are present, the soil being described can be termed homogeneous if the description of the lenses is included
- Prismatic or Columnar – particles arranged about a vertical line, ped is bounded by planar, vertical faces that accommodate adjoining faces; prismatic has a flat top; columnar has a rounded top
- Platy – particles are arranged about a horizontal plane

8.1.5.4 Other Features

- Mottled – soil that appears to consist of material of two or more colors in blotchy distribution
- Fissured – breaks along definite planes of fracture with little resistance to fracturing (determined by applying moderate pressure to sample using thumb and index finger)
- Slickensided – fracture planes appear polished or glossy, sometimes striated (parallel grooves or scratches)

8.1.6 **Development of Soil Description**

Develop standard soil descriptions according to the following examples. There are three principal categories under which all soil can be classified. They are described below.

8.1.6.1 Coarse-grained Soil

Coarse-grained soil is divided into sands and gravels. A soil is classified as a sand if over 50 percent of the coarse fraction is “sand-sized.” It is classified as a gravel if over 50 percent of the coarse fraction is composed of “gravel-sized” particles.

The written description of a coarse-grained soil shall contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); grain size of coarse fraction; Munsell color and color number; moisture content; relative density; sorting; angularity; other features, such as stratification (sedimentary structures) and cementation, possible formational name, primary USCS classification, secondary USCS classification (when necessary), and approximate percentages of minor constituents (i.e., sand, gravel, shell fragments, rip-up clasts) in parentheses.

Example: POORLY-SORTED SAND WITH SILT, medium- to coarse-grained, light olive gray, 5Y 6/2, saturated, loose, poorly sorted, subrounded clasts, SW/SM (minor silt with approximately 20 percent coarse-grained sand-sized shell fragments, and 80 percent medium-grained quartz sand, and 5 percent to 15 percent ML).

8.1.6.2 Fine-grained Soil

Fine-grained soil is further subdivided into clays and silts according to its plasticity. Clays are rather plastic, while silts have little or no plasticity.

The written description of a fine-grained soil should contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); Munsell color; moisture content; consistency; plasticity; other features, such as stratification, possible formation name, primary USCS classification, secondary USCS classification (when necessary), and the percentage of minor constituents in parentheses.

Example: SANDY LEAN CLAY, dusky red, 2.5 YR 3/2, moist, firm, moderately plastic, thinly laminated, CL (70 percent fines, 30 percent sand, with minor amounts of disarticulated bivalves [about 5 percent]).

8.1.6.3 Organic Soil

For highly organic soil, describe the types of organic materials present as well as the type of soil constituents present using the methods described above. Identify the soil as an organic soil, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soil usually has a dark

brown to black color and may have an organic odor. Often, organic soils will change color, (e.g., from black to brown) when exposed to air. Some organic soils will lighten in color significantly when air-dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

8.2 Example: ORGANIC CLAY, black, 2.5Y, 2.5/1, wet, soft, low plasticity, organic odor, OL (100 percent fines), weak reaction to HCl.

8.3 Rock Classification

The purpose of rock classification is to thoroughly describe the physical and mineralogical characteristics of a specimen and to classify it according to an established system. The generalized rock classification system described below was developed because, unlike the USCS for soils, there is no universally accepted rock classification system. In some instances, a more detailed and thorough rock classification system may be appropriate. Any modifications to this classification system, or the use of an alternate classification system should be considered during preparation of the site work plan. Both the DO Manager and the QA Manager or Technical Director must approve any modifications to this classification system, or the use of another classification system.

Describing rock specimens on a common basis is essential so that rocks described by different site geologists are comparable. Site geologists describing rock specimens as a part of investigative activities must use the classification system described herein, or if necessary, another more detailed classification system. Use of a common classification system provides the most useful geologic database for all present and future subsurface investigations and remedial activities.

In order to provide a more consistent rock classification between geologists, a rock classification template has been designated as shown in **Error! Reference source not found.**. The template includes classification of rocks by origin and mineralogical composition. When classifying rocks, all site geologists shall use this template.

The site geologist shall describe the rock specimen and record the description in a boring log or logbook. The items essential for classification include (i.e., metamorphic foliated):

- Classification Name (i.e., schist)
- Color
- Mineralogical composition and percent
- Texture/Grain size (i.e., fine-grained, pegmatitic, aphlitic, glassy)
- Structure (i.e., foliated, fractured, lenticular)
- Rock Quality Designation (sum of all core pieces greater than two times the diameter of the core divided by the total length of the core run, expressed as a percentage)
- Classification symbol (i.e., MF)

Example: Metamorphic foliated schist: Olive gray, 5Y, 3/2, Garnet 25 percent, Quartz 45 percent, Chlorite 15 percent, Tourmaline 15 percent, Fine-grained with Pegmatite garnet, highly foliated, slightly wavy, MF.

9.0 Quality Control and Assurance

None

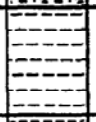
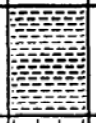
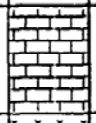
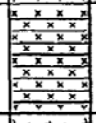
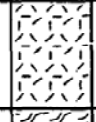
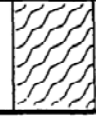
DEFINITION OF TERMS					
PRIMARY DIVISIONS				SYMBOLS	SECONDARY DIVISIONS
SEDIMENTARY ROCKS	Clastic Sediments	CONGLOMERATE		CG	Coarse-grained Clastic Sedimentary Rock types including: Conglomerates and Breccias
		SANDSTONE		SS	Clastic Sedimentary Rock types including: Sandstone, Arkose and Greywacke
		SHALE		SH	Fine-grained Clastic Sedimentary Rock types including: Shale, Siltstone, Mudstone and Claystone
	Chemical Precipitates	CARBONATES		LS	Chemical Precipitates including: Limestone, Crystalline Limestone, Fossiliferous Limestone Micrite and Dolomite
		EVAPORITES		EV	Evaporites including: Anhydrite, Gypsum, Halite, Travertine and Caliche
IGNEOUS ROCKS		EXTRUSIVE (Volcanic)		IE	Volcanic Rock types including: Basalt, Andesite, Rhyolite, Volcanic Tuff, and Volcanic Breccia
		INTRUSIVE (Plutonic)		II	Plutonic Rock types including: Granite, Diorite and Gabbro
METAMORPHIC ROCKS		FOLIATED		MF	Foliated Rock types including: Slate, Phyllite, Schist and Gneiss
		NON-FOLIATED		MN	Non-foliated Rock types including: Metaconglomerate, Quartzite and Marble

Figure 8-4: Rock Classification System

10.0 Data and Records Management

10.1 Document soil classification information collected during soil sampling onto the field boring logs, field trench logs, and into the field notebook. Copies of this information shall be sent to the **DO Manager** for the project files.

10.2 Field notes will be kept during coring activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. The information pertinent to soil classification activities includes chronology of events, sample locations (x,y,z), time/date, sampler name, methods (including type of core liner/barrel, if applicable), sampler penetration and acceptability, sample observations, and the times and type of equipment decontamination. Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

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Huang, Walter T. 1962. *Petrology*. New York: McGraw-Hill Book Company.

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Direct Push Sampling Techniques

Procedure 3-17

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) provides guidance on the use of direct push techniques.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV).

1.3 This procedure shall serve as management-approved professional guidance and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Delivery Order (DO) Manager and the Quality Assurance (QA) Manager or Technical Director, and documented.

1.4 If there are procedures whether it be from AECOM-Tidewater JV, state and/or federal that are not addressed in this SOP and are applicable to direct push sampling then those procedures may be added as an appendix to the project specific SAP.

2.0 Safety

2.1 Field personnel shall perform work in accordance with the site-specific health and safety plan (HASP). During monitoring well installation, subcontractors in direct contact with potentially contaminated media shall wear the proper personal protective equipment (PPE) as outlined in the site-specific health and safety plan. Failure to comply will result in disciplinary action.

2.2 If circumstances warrant, a real-time immediate response instrument, such as a Miniram Dust Monitor, organic vapor analyzer, HNu, Thermo, Draeger or Sensidyne tubes, or explosimeter, should be used to monitor the work area. When real/time instrument response exceeds the permissible exposure limit, personnel shall don the appropriate PPE and alternate control measures to ensure personnel safety. If safe control measures are not achievable, field activities shall be discontinued immediately. Company-specific HASPs offer guidelines on air surveillance and on selection of PPE. In addition, the site-specific HASP includes an air monitoring program and suggested PPE.

2.3 In addition to the aforementioned precautions and depending upon the type of contaminant expected, employ the following safe work practices:

Particulate or Metal Compounds

1. Avoid skin contact and/or incidental ingestion of soil.
2. Wear protective clothing, safety-toed boots, gloves, safety glasses, and hearing protection as warranted.

VOCs

1. Avoid breathing constituents venting from holes by approaching upwind, and/or by use of respiratory protection.
2. Pre-survey the area with a flame ionization detector (FID) or photoionization detector (PID) prior to sampling.
3. If monitoring results indicate organic vapors that exceed action levels as specified in the site-specific HASP, sampling activities may need to be conducted in Level C protection. At a minimum, skin protection will be required by use of gloves and Tyvek or other media that is protective against the media being encountered.

Flammable or Explosive Conditions

1. Monitor explosive gases as continuously as possible using an explosimeter and oxygen meter.
2. Place all ignition sources upwind or crosswind of the borehole.
3. If explosive gases exceed the designated action levels as specified in the site-specific HASP, cease operations and evaluate conditions.

Physical Hazards Associated With Soil Sampling

1. To avoid possible back strain associated with sample collection, use the large muscles of the legs, not the back, when retrieving soil samplers.
2. Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
3. To avoid slip/trip/fall hazards, be wary of open trenches, pits, or holes.
4. Be aware of restricted mobility due to PPE.
5. To avoid hand, wrist, arm, shoulder, and back trauma due to the use of slide hammers or hand augers, rotate sampling among field personnel

3.0 Terms and Definitions

3.1 Direct push techniques are methods for subsurface sampling or monitoring that involve the application of downward pressure (usually supplied through hydraulic means) without the benefit of cutting tool rotation to enter soil. A variety of systems are available under several trade names, such as GeoProbe®. Equipment may be skid-mounted, trailered, or mounted directly on the frame of a vehicle.

4.0 Interferences

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The **DO Manager** is responsible for ensuring that these standard direct push technique procedures are followed and that a qualified individual conducts or supervises the projects. A qualified individual for subsurface sampling or monitoring using direct push techniques is defined as a person with a degree in geology, hydrogeology, or geotechnical/civil engineering with at least 1 year of experience supervising soil boring construction using conventional drilling or direct push techniques. The DO Manager or designee is responsible for ensuring that all personnel involved in direct push sampling techniques shall have the appropriate education, experience, and training to perform their assigned tasks as specified in the DoD Environmental Field Sampling Handbook (DOD 2013).

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all field personnel follow these procedures.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

5.2.5 The Field Personnel and/or Field Manager is responsible for directly supervising the direct push sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

In addition to those materials provided by the subcontractor, the project **Field Manager/Field Personnel** will require:

- Boring Logs;
- Spoons or scoops;
- Sample kit (bottles, labels, custody records and tape, cooler, ice), if laboratory analysis is required;
- Sample collection pan;
- Folding rule or tape measure;
- Plastic sheeting;
- Utility knife;
- Equipment decontamination materials (as described in SOP 3-06, *Equipment Decontamination*);
- Health and safety equipment (as required by HASP); and
- Field project notebook/pen.

7.0 Procedure

Direct push techniques may be used as a cost-effective alternative to conventional drilling techniques for obtaining subsurface soil and groundwater samples and for monitoring subsurface conditions.

7.1 Method Selection

Base the decision to use direct push techniques on: (1) their ability to achieve the required information at the required level of quality control and (2) their cost-effectiveness compared to conventional drilling methods. Major limitations of direct push techniques are their inability to penetrate rock or cobbles and a shallow maximum depth of penetration. The capabilities of direct push systems vary significantly among vendors. Consider these differences in capabilities when evaluating the method for a subsurface exploration program.

Use direct push techniques to obtain groundwater samples for confirmatory analyses only if the screen placement method protects the screen from clogging during installation and allows the installation of a sand-pack around the exterior of the well screen.

7.2 Inspection of Equipment

Inspect direct push equipment prior to use for signs of fluid leakage, which could introduce contaminants to the soil. If, at any time during equipment operation, fluid is observed leaking from the rig, cease operations and immediately repair or contain the leak. Collect, containerize, and label soil and other materials affected by the leak for proper disposal (see SOP 3-05, *IDW Management*).

7.3 Preparation of Work Site

Inspect the work site prior to commencing operations to ensure that no overhead hazards exist that could impact the direct push equipment, and the work area should cleared and/or marked by the local underground utility locating service (e.g., DigSafe). In addition, clear locations planned for subsurface exploration using either geophysical methods and/or hand excavate locations to a depth of 2 to 3 feet prior to soil penetration, unless it is certain (by virtue of subsurface clearing activities) that no utilities or other hazardous obstructions will be encountered in the first 2 to 3 feet. Hand excavation may be waived when it is not practical.

Locate the direct push rig so that it is downslope from the penetration point, if the work is to be performed on a grade. Locate the rig downwind or crosswind of the penetration point, if possible. Cover the area surrounding, and in the vicinity of, the penetration point with plastic. Establish required exclusion zones using plastic tape or cones to designate the various areas.

7.4 Equipment Decontamination

To avoid cross-contamination, thoroughly decontaminate equipment used for direct push exploration and sampling as described in SOP 3-06, *Equipment Decontamination*. Decontaminate sampling tools and downhole equipment between each sampling event and between penetration points. At a minimum,

steam clean or wash and rinse the equipment. Collect, containerize, and label all wash and rinse water for proper disposal. Clean equipment (e.g., drive rods and samplers) shall not come into contact with contaminated soils or other contaminated materials. Keep equipment on plastic or protect it in another suitable fashion. Store push rods and other equipment removed from a hole on plastic sheeting until properly decontaminated.

7.5 Soil Sampling

This SOP assumes that the subcontractor will perform sampling; therefore, detailed procedures regarding sample acquisition are not provided. Vendors of direct push equipment offer a variety of sampling systems designed specifically for their equipment. Both continuous and discreet soil samples may be obtained using sampling equipment similar to that described in Procedure 3-21, *Surface and Subsurface Soil Sampling*. The preferred methods for soil sampling using direct push techniques use brass or stainless steel split-tube samplers that are driven through the horizon to be sampled. Use plastic sample tubes (e.g., Macro-Core Samplers) only for screening purposes or, in the case of confirmatory sampling, if samples will not be analyzed for volatile organic compounds (VOCs) or semivolatile organic compounds (SVOCs).

7.6 Groundwater Sampling

Direct push vendors offer numerous methods for obtaining groundwater samples. Key differences among methods involve: (1) the maximum well diameter achievable; (2) the ability to protect the well screen from exposure to contaminated overburden soils during installation; (3) the ability to install packing around the screen; (4) flexibility in the size, materials of construction, and design of well screens; and (5) the ability to convert sampling points into permanent monitoring wells. The limitations and abilities of a given system must be thoroughly understood and matched to the needs of the project before committing to the collection of groundwater samples using direct push techniques.

Use direct push techniques only to collect screening samples unless it is confirmed that the system:

1. Effectively protects the well screen from exposure to contaminated overburden soils during installation
2. Allows the installation of effective packing around the well screen
3. Allows the well screen to be effectively sealed against the downward infiltration of overlying groundwater or surface precipitation
4. Is constructed of materials compatible with the intended sampling and analysis goals of the project
5. Allows the use of a well screen properly sized and slotted for the needs of the project

Additional information on the collection of groundwater samples can be found in SOP 3-14 Monitoring Well Sampling.

It is the responsibility of the **DO Manager** to evaluate and determine the appropriateness of direct push systems prior to committing to their use on any project involving groundwater sampling. As part of this evaluation, it is recommended to obtain concurrence from regulatory authorities in advance for the method selection.

7.7 Borehole Abandonment

Methods for abandoning boreholes created with direct push systems will vary among vendors. Coordinate the desired method for abandonment with the vendor in the planning stages of the project to ensure proper abandonment.

Some direct push boreholes will close naturally as the drive rods and sampling tools are withdrawn. This may occur in loose, unconsolidated soils, such as sands. Close all boreholes using one of the procedures described in this procedure, unless natural caving precludes such closure.

The three methods for closing direct push boreholes are:

1. Add granulated or pelletized bentonite and hydrate in layers, proceeding from the bottom of the hole to the surface.
2. Pour premixed cement/water (or cement/water/bentonite) mixture into the hole.

3. Fill the entire hole with granular or pelletized bentonite and hydrate by means of a previously emplaced water tube that is gradually withdrawn as water is supplied to the bentonite.

The second method is recommended. For shallow holes less than 10 feet in depth, pour a cement/water/bentonite mix directly into the opening using a funnel. For deeper holes, use a conductor (tremie) pipe to carry the grout mix to the far reaches of the borehole. Lower the conductor pipe to within 2 inches of the bottom and gradually withdraw it as grout is added, keeping the lower end of the pipe submerged in grout at all times.

The recommended grout mixture for well abandonment is 7 to 9 gallons of water per 94-pound bag of Portland cement, with 3 percent to 5 percent by weight of powdered bentonite added to the mixture. Commercial products, such as Volcay are acceptable with pre-approval of the **DO Manager**.

Seal boreholes to within 0.5 to 2.0 feet of the surface. Inspect the abandoned borehole after 24 hours to ensure that grout shrinkage does not occur. If significant shrinkage has occurred, re-grout the borehole. Fill the remaining portion of the hole with local topsoil or appropriate paving materials.

8.0 Quality Control and Assurance

8.1 Collection of representative samples will be ensured through adherence to the procedures in this SOP and the sampling strategy outlined in the SAP. The field quality control samples identified in the SAP must be collected. These samples may include field duplicates, equipment rinsate blanks, trip blanks, and matrix spike/matrix spike duplicates

9.0 Records, Data Analysis, Calculations

9.1 Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Boring logs;
- Field logbook;
- Sample collection records;
- Chain-of-custody forms; and
- Shipping labels.

9.2 Boring logs (Attachment 1) will provide visual and descriptive information for samples collected at each soil boring and are often the most critical form of documentation generated during a soil sampling program.

9.3 The field logbook is kept as a general log of activities and should not be used in place of the boring log.

9.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

9.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

10.0 Attachments or References

10.1 Attachment 1 – Boring Log

10.2 *DoD Environmental Field Sampling Handbook,* Revision 1.0. April 2013.

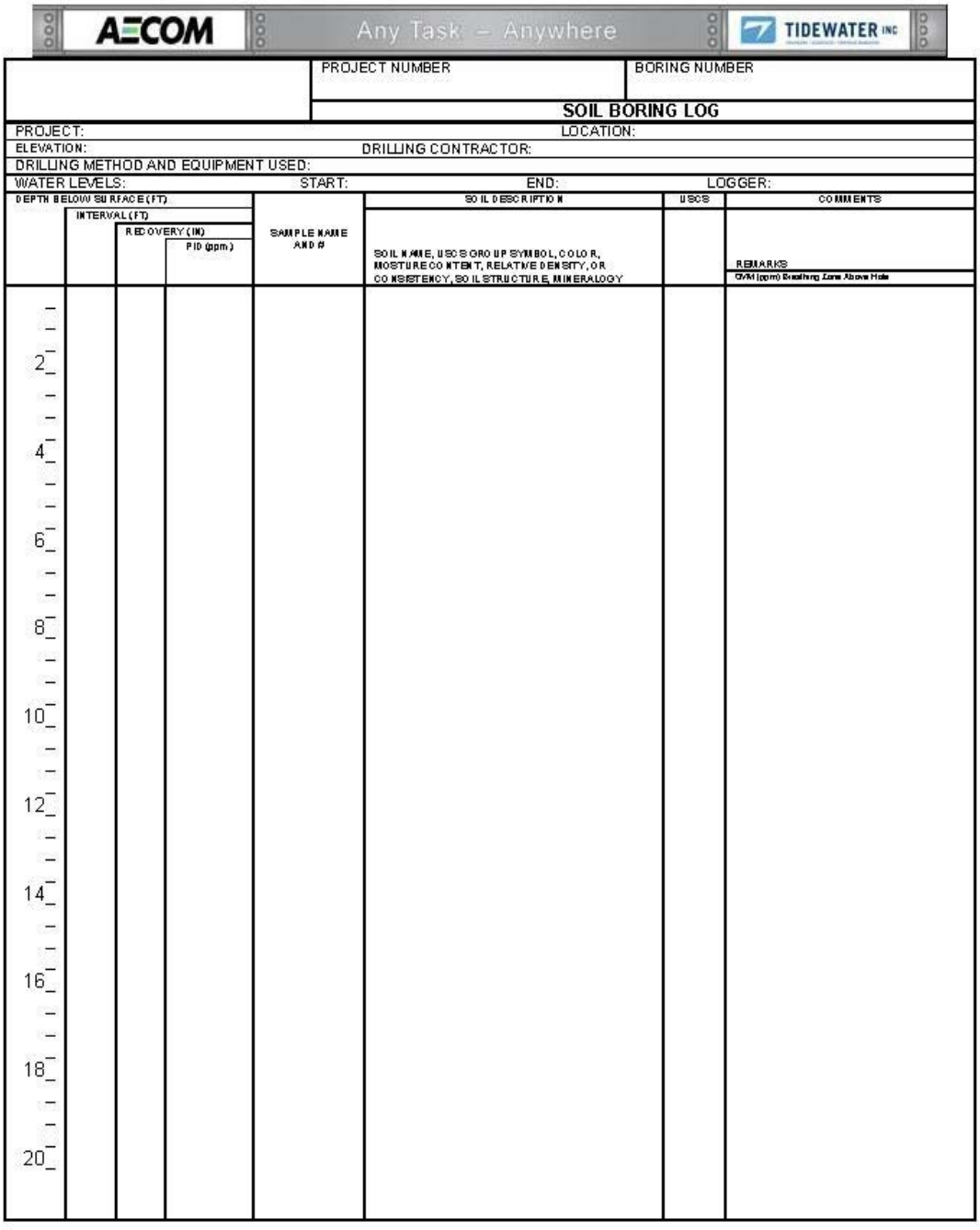
10.3 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

10.4 SOP 3-05, *IDW Management.*

10.5 SOP 3-06, *Equipment Decontamination.*

10.6 SOP 3-21, *Surface and Subsurface Soil Sampling.*

Attachment 1
Boring Log



AECOM Any Task – Anywhere TIDEWATER INC

PROJECT NUMBER	BORING NUMBER

SOIL BORING LOG

PROJECT: LOCATION:

ELEVATION: DRILLING CONTRACTOR:

DRILLING METHOD AND EQUIPMENT USED:

WATER LEVELS: START: END: LOGGER:

DEPTH BELOW SURFACE (FT)	INTERVAL (FT)	RECOVERY (IN)	PID (ppm)	SAMPLE NAME AND #	SOIL DESCRIPTION SOIL NAME, USCS GROUP SYMBOL, COLOR, MOISTURE CONTENT, RELATIVE DENSITY, OR CONSISTENCY, SOIL STRUCTURE, MINERALOGY	USCS	COMMENTS REMARKS OVM (ppm) Breathing Zone Above Hole
2							
4							
6							
8							
10							
12							
14							
16							
18							
20							

Headspace Screening for Total VOCs

Procedure 3-19

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the basic techniques for using headspace analysis to screen for volatile organics in contaminated soils using a portable Photo Ionization Detector (PID) or Flame Ionization Detector (FID).

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Delivery Order (DO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**. Note that headspace screening usually requires Level D personal protection unless there is a potential for airborne exposure to site contaminants. Under circumstances where potential airborne exposure is possible respiratory protective equipment may be required based on personal air monitoring results. Upgrades to Level C will be coordinated with the Site Safety Officer (SSO) or **DO Manager**.

2.2 Health and safety hazards and corresponding precautions include, but are not limited to, the following:

2.2.1 Dermal contact with contaminated soil. Personnel should treat all soil as potentially contaminated and wear chemically impervious gloves. Minimize skin contact with soil by using sampling instruments such as stainless steel spades or spoons. Do not touch any exposed skin with contaminated gloves.

2.2.2 Inhalation hazards. Appropriate air monitoring should be conducted to ensure that organic vapor concentrations in the breathing zone do not exceed action levels as specified in the Site-Specific HASP. When ambient temperatures are low enough to require warming samples using the vehicle heater, the vehicle's windows should be opened enough to prevent the build-up of any organic vapors. Use the PID or FID to verify the airborne concentrations in the vehicle remain below applicable action levels. Note that many volatile organic compounds (VOCs) are flammable and all precautions must be observed to eliminate any potential ignition sources.

2.2.3 Shipping limitations. Follow applicable regulations when shipping FID/PID equipment. When shipping an FID by air, the hydrogen tank must be bled dry. Calibration gas canisters are considered dangerous goods and must be shipped according to IATA and DOT regulations. Consult your EHS Coordinator and check with your shipping company to determine the correct shipping procedures

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to

minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The DO Manager is responsible for ensuring that the collection of headspace readings comply with this procedure. The DO Manager is responsible for ensuring that all personnel involved in the collection of headspace readings shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all headspace readings are conducted according to this procedure as well as verifying that the PID/FID is in proper operating condition prior to use and for implementing the calibration.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

6.1 The following materials must be on hand in good operating condition and/or in sufficient quantity to ensure that proper field analysis procedures may be followed:

- Calibrated PID/FID instrument;
- Top-sealing "Zip-Loc" type plastic bags – or – 16 ounces of soil or "mason-" type glass jars and aluminum foil;
- Project field book and/or boring logs;
- Personal Protective Equipment (PPE) as specified in the project HASP; and
- Material Safety Data Sheets (MSDSs) for any chemicals or site-specific contaminants.

7.0 Procedure

7.1 **Preparation**

Review available project information to determine the types of organic vapors that will likely be encountered to select the right instrument. The two basic types of instruments are FIDs and PIDs.

FIDs work well with organic compounds that have relatively lightweight molecules, but may have problems detecting halogenated compounds or heavier organic compounds; FIDs can detect methane for example. Since the FID uses a flame to measure organic compounds, ensure that work is conducted in an atmosphere, which is free of combustible vapors. If ambient temperatures are below 40°F, the flame of the FID may be difficult to light.

When using a PID, select an instrument that can measure the ionization potential of the anticipated contaminants of concern. PIDs work well with a range of organic compounds and can detect some halogenated hydrocarbons; PIDs cannot detect methane. The correct ultraviolet (UV) light bulb must be selected according to the types of organic vapors that will likely be encountered. The energy of the UV light must equal or exceed the ionization potential of the organic molecules that the PID will measure. The NIOSH Pocket Guide to Chemical Hazards is one source for determining ionization potentials for different chemicals. Bulbs available for PIDs include 9.4 eV, 10.6 (or 10.2) eV, and 11.7 eV bulbs. The 10.6 eV bulb is most commonly used as it detects a fairly large range of organic molecules and does not burn out as easily as the 11.7 eV bulb. The 9.4 eV bulb is the most rugged, but detects only a limited range of compounds. Under very humid or very cold ambient conditions, the window covering the UV light may fog up, causing inaccurate readings. Ask the **SSO** about correction factors when high humidity conditions exist.

After selecting the correct instrument, calibrate the PID/FID according to the manufacturer's instructions. Record background/ambient levels of organic vapors measured on the PID/FID after calibration and make sure to subtract the background concentration (if any) from your readings. Check the PID/FID readings against the calibration standard every 20 readings or at any time when readings are suspected to be inaccurate, and recalibrate, if necessary. Be aware that, after measuring highly contaminated soil samples, the PID/FID may give artificially high readings for a time.

7.2 Top-Sealing Plastic Bag

Place a quantity of soil in a top-sealing plastic bag and seal the bag immediately. The volume of soil to be used should be determined by the **DO Manager** or **Field Manager**. The volume of soil may vary between projects but should be consistent for all samples collected for one project. Ideally, the bag should be at least 1/10th-filled with soil and no more than half-filled with soil. Once the bag is sealed, shake the bag to distribute the soil evenly. If the soil is hard or clumpy, use your fingers to gently work the soil (through the bag) to break up the clumps. Do not use a sampling instrument or a rock hammer since this may create small holes in the plastic bag and allow organic vapors to escape. Alternatively, the sample may be broken up before it is placed in the bag. Use a permanent marker to record the following information on the outside of the bag:

- Site identification information (i.e., borehole number);
- Depth interval; and
- Time the sample was collected. For example: "SS-12, 2-4 ft, @1425".

Headspace should be allowed to develop before organic vapors are measured with a PID/FID. The amount of time required for sufficient headspace development will be determined by the project-specific sampling plan and the ambient temperature. Equilibration time should be the same for all samples to allow an accurate comparison of organic vapor levels between samples. However, adjustments to equilibration times may be necessary when there are large variations in ambient temperature from day to day. When ambient temperatures are below 32°F, headspace development should be within a heated building or vehicle. When heating samples, be sure there is adequate ventilation to prevent the build-up or organic vapors above action levels.

Following headspace development, open a small opening in the seal of the plastic bag. Insert the probe of a PID/FID and seal the bag back up around the probe as tightly as possible. Alternatively, the probe can be inserted through the bag to avoid loss of volatiles. Since PIDs and FIDs are sensitive to moisture, avoid touching the probe to the soil or any condensation that has accumulated inside of the bag. Since the PID/FID consumes organic vapors, gently agitate the soil sample during the reading to release fresh organic vapors from the sample. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case, headspace data should be discounted. Record the highest reading on the field form or in the field notebook as described in Section 9.

7.3 **Jar and Aluminum Foil (Alternate Method)**

Half-fill a clean glass jar with the soil sample to be screened. Quickly cover the jar's opening with one to two sheets of clean aluminum foil and apply the screw cap to tightly seal the jar. Allow headspace development for at least ten minutes. Vigorously shake the jar for 15 seconds, both at the beginning and at the end of the headspace development period. Where ambient temperatures are below 32°F (0°C), headspace development should be within a heated area. When heating samples, be sure there is adequate ventilation to prevent the build-up of organic vapors above action levels.

Subsequent to headspace development, remove the jar lid and expose the foil seal. Quickly puncture the foil seal with the instrument sampling probe, to a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particulates. As an alternative, use a syringe to withdraw a headspace sample, and then inject the sample into the instrument probe or septum-fitted inlet. This method is acceptable contingent upon verification of methodology accuracy using a test gas standard. Following probe insertion through the foil seal or sample injection to probe, record the highest meter response on the field form or in the field notebook. Using foil seal/probe insertion method, maximum response should occur between two and five seconds. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case, headspace data should be discounted.

8.0 Quality Control and Assurance

Quality Assurance/Quality Control (QA/QC) will include the collection of duplicate samples. In general, one duplicate will be collected per 20 samples. Organic vapor concentrations measured in the primary and duplicate samples should be similar within plus or minus 20 percent. The frequency of headspace duplicate collection will be determined by the project manager/task manager. The PID/FID instrument must be calibrated according to the manufacturer's instructions before beginning screening, and checked or recalibrated every 20 analyses or when readings are suspected to be inaccurate. Record ambient organic vapor levels in the field notebook and on the field form. Periodically check ambient organic vapor levels. If ambient levels have changed more than 20 percent, recalibrate the PID/FID. Make sure readings are not collected near a vehicle exhaust or downwind of a drill rig exhaust. If grossly contaminated soil is encountered, decontaminate sampling instruments between samples and/or change contaminated gloves to avoid cross contaminating less contaminated samples.

9.0 Records, Data Analysis, Calculations

9.1 All data generated (results and duplicate comparisons) will be recorded in the field notebook and/or on the field form. Any deviation from the outlined procedure will also be noted. Field conditions (ambient temperature, wind, etc.) should also be recorded in the field notebook.

9.2 Readings may be recorded in a field notebook, on a boring log, or on an appropriate form specific to the project. The form should include the following information:

- When the PID/FID was calibrated (date/time) and calibration standard used;
- Background/ambient concentrations measured after PID/FID calibration;
- Location of sample (i.e., bore-hole number);
- Depth interval of sample measured;
- Lithology of material measured; and
- PID/FID reading and units of measure.

9.3 Note that if PID/FID measurements are recorded on a boring log, it is not necessary to duplicate information in the column where the PID/FID readings are recorded (e.g., borehole number, depth interval, lithology type).

9.4 All documentation will be stored in the project files and retained following completion of the project.

10.0 Attachments or References

SOP 3-20 Operation and Calibration of a Photoionization Detector

Operation and Calibration of a Photoionization Detector

Procedure 3-20

1.0 Purpose and Scope

1.1 Purpose and Applicability

1.1.1 This standard operating procedure (SOP) describes the procedures that will be followed by field staff for operation and calibration of a photoionization detector (PID). The PID is primarily used by AECOM-Tidewater JV personnel for safety and survey monitoring of ambient air, determining the presence of volatiles in soil and water, and detecting leakage of volatiles.

1.1.2 PIDs routinely used by field personnel include the Photovac Microtip, Thermoelectron 580EZ, and MiniRAE 2000 or 3000. Personnel responsible for using the PID should first read and thoroughly familiarize themselves with the instrument instruction manual.

1.2 Principle of Operation

1.2.1 The PID is a non-specific vapor/gas detector. The unit generally consists of a hand-held probe that houses a PID, consisting of an ultraviolet (UV) lamp, two electrodes, and a small fan which pulls ambient air into the probe inlet tube. The probe is connected to a readout/control box that consists of electronic control circuits, a readout display, and the system battery. Units are available with UV lamps having an energy from 9.5 electron volts (eV) to 11.7 eV.

1.2.2 The PID analyzer measures the concentration of trace gas present in the atmosphere by photoionization. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and become a positive ion. This will occur when the ionization potential of the molecule (in electron volts (eV)) is less than the energy of the photon. The source of photons is an ultraviolet lamp in the probe unit. Lamps are available with energies ranging from 9.5 eV to 11.7 eV. All organic and inorganic vapor/gas compounds having ionization potentials lower than the energy output of the UV lamp are ionized and the resulting potentiometric change is seen as a positive reading on the unit. The reading is proportional to the concentration of organics and/or inorganics in the vapor.

1.2.3 Sample gases enter the probe through the inlet tube and enter the ion chamber where they are exposed to the photons emanating from the UV lamp. Ionization occurs for those molecules having ionization potentials near to or less than that of the lamp. A positive- biased polarizing electrode causes these positive ions to travel to a collector electrode in the chamber. Thus the ions create an electrical current which is amplified and displayed on the meter. This current is proportional to the concentration of trace gas present in the ion chamber and to the sensitivity of that gas to photoionization.

1.2.4 In service, the analyzer is first calibrated with a gas of known composition equal to, close to, or representative of that to be measured. Gases with ionization potentials near to or less than the energy of the lamp will be ionized. These gases will thus be detected and measured by the analyzer. Gases with ionization potentials greater than the energy of the lamp will not be detected. The ionization potentials of the major components of air, i.e., oxygen, nitrogen, and carbon dioxide, range from about 12.0 eV to 15.6 eV and are not ionized by any of the lamps available. Gases with ionization potentials near to or slightly higher than the lamp are partially ionized, with low sensitivity.

1.3 Specifications

1.3.1 Refer to the manufacturer's instructions for the technical specifications of the instrument being used. The operating concentration range is typically 0.1 to 2,000 ppm isobutylene equivalent.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Delivery Order (DO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.

2.2 Only PIDs stamped Division I Class I may be used in explosive atmospheres. Refer to the project HASP for instructions pertaining to instrument use in explosive atmospheres.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The DO Manager is responsible for ensuring that the operation and calibration activities comply with this procedure. The DO Manager is responsible for ensuring that all personnel involved in the operation and calibration shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all operation and calibration activities are conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

- Calibration Gas: Compressed gas cylinder of isobutylene in air or similar stable gas mixture of known concentration. The selected gas should have an ionization potential similar to that of the vapors to be monitored, if known. The concentration should be at 50-75% of the range in which the instrument is to be calibrated;

- Regulator for calibration gas cylinder;
- Approximately 6 inches of Teflon® tubing;
- Tedlar bag (optional);
- Commercially-supplied zero grade air (optional);
- "Magic Marker" or “Sharpie” or other waterproof marker;
- Battery charger;
- Moisture traps;
- Spare lamps;
- Manufacturer’s instructions; and
- Field data sheets or logbook/pen.

7.0 Procedure

7.1 Preliminary Steps

7.1.1 Preliminary steps (battery charging, check-out, calibration, maintenance) should be conducted in a controlled or non-hazardous environment.

7.2 Calibration

7.2.1 The PID must be calibrated in order to display concentrations in units equivalent to ppm. First a supply of zero air (ambient air or from a supplied source), containing no ionizable gases or vapors is used to set the zero point. A span gas, containing a known concentration of a photoionizable gas or vapor, is then used to set the sensitivity.

7.2.2 Calibrate the instrument according to the manufacturer’s instructions. Record the instrument model and identification number, the initial and adjusted meter readings, the calibration gas composition and concentration, and the date and the time in the field records.

7.2.3 If the calibration cannot be achieved or if the span setting resulting from calibration is 0.0, then the lamp must be cleaned (Section 7.4).

7.3 Operation

7.3.1 Turn on the unit and allow it to warm up (minimum of 5 minutes). Check to see if the intake fan is functioning; if so, the probe will vibrate slightly and a distinct sound will be audible when holding the probe casing next to the ear. Also, verify on the readout display that the UV lamp is lit.

7.3.2 Calibrate the instrument as described in Section 7.2, following the manufacturer’s instructions. Record the calibration information in the field records.

7.3.3 The instrument is now operational. Readings should be recorded in the field records.

7.3.4 When the PID is not being used or between monitoring intervals, the unit may be switched off to conserve battery power and UV lamp life; however, a “bump” test should be performed each time the unit is turned on and prior to taking additional measurements. To perform a bump test, connect the outlet tubing from a Tedlar bag containing a small amount of span gas to the inlet tubing on the unit and record the reading. If the reading is not within the tolerance specified in the project plan, the unit must be recalibrated.

7.3.5 At the end of each day, recheck the calibration. The check will follow the same procedures as the initial calibration (Section 7.2) except that no adjustment will be made to the instrument. Record the information in the field records.

7.3.6 Recharge the battery after each use (Section 7.4).

7.3.7 When transporting, ensure that the instrument is packed in its stored condition in order to prevent damage.

7.4 **Routine Maintenance**

7.4.1 Routine maintenance associated with the use of the PID includes charging the battery, cleaning the lamp window, replacing the detector UV lamp, replacing the inlet filter, and replacing the sample pump. Refer to the manufacturer's instructions for procedures and frequency.

7.4.2 All routine maintenance should be performed in a non-hazardous environment.

7.5 **Troubleshooting Tips**

7.5.1 One convenient method for periodically confirming instrument response is to hold the sensor probe next to the tip of a magic marker. A significant reading should readily be observed.

7.5.2 Air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings.

7.5.3 A fogged or dirty lamp, due to operation in a humid or dusty environment, may cause erratic or fluctuating readings. The PID should never be operated without the moisture trap in place.

7.5.4 Moving the instrument from a cool or air-conditioned area to a warmer area may cause moisture to condense on the UV lamp and produce unstable readings.

7.5.5 A zero reading on the meter should not necessarily be interpreted as an absence of air contaminants. The detection capabilities of the PID are limited to those compounds that will be ionized by the particular probe used.

7.5.6 Many volatile compounds have a low odor threshold. A lack of meter response in the presence of odors does not necessarily indicate instrument failure.

7.5.7 When high vapor concentrations enter the ionization chamber in the PID the unit can become saturated or "flooded". Remove the unit to a fresh air environment to allow the vapors to be completely ionized and purged from the unit.

8.0 Quality Control and Assurance

8.1 The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Sampling and Analysis Plan (SAP), hereafter referred to as the project plan.

8.2 Calibration of the PID will be conducted at the frequency specified in the project plan. In the absence of project-specific guidance, calibration will be performed at the beginning of each day of sampling and will be checked at the end of the sampling day or whenever instrument operation is suspect. The PID will sample a calibration gas of known concentration. The instrument must agree with the calibration gas within ±10%. If the instrument responds outside this tolerance, it must be recalibrated.

8.3 Checks of the instrument response (Section 7.5) should be conducted periodically and documented in the field records.

9.0 Records, Data Analysis, Calculations

Safety and survey monitoring with the PID will be documented in a bound field logbook, or on standardized forms, and retained in the project files. The following information is to be recorded:

- Project name and number;
- Instrument manufacturer, model, and identification number;

- Operator's signature;
- Date and time of operation;
- Calibration gas used;
- Calibration check at beginning and end of day (meter readings before adjustment);
- Span setting after calibration adjustment;
- Meter readings (monitoring data obtained);
- Instances of erratic or questionable meter readings and corrective actions taken; and
- Instrument checks and response verifications – e.g., battery check, magic marker response (Section 7.5) or similar test.

10.0 Attachments or References

United States Environmental Protection Agency. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM). USEPA, Region 4, SESD, Enforcement and Investigations Branch, Athens, GA. November 2001.

Surface and Subsurface Soil Sampling Procedures

Procedure 3-21

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures for soil sampling. The procedure includes surface and subsurface sampling by various methods using hand auguring, test pit, direct-push, and split-spoon equipment.

1.2 The procedure includes soil sampling for volatile organic compounds (VOCs). For project specific information (e.g. sampling depths, equipment to be used, and frequency of sampling), refer to the Sampling and Analysis Plan (SAP), which takes precedence over these procedures. Surface soil sampling, typically considered to be up to two feet below ground surface by EPA standards, is typically accomplished using hand tools such as shovels or hand augers. Test pit samples are considered subsurface samples, although normally collected via hand tools similar to surface soil sampling or by excavation machinery. Direct-push and split-spoon sampling offer the benefit of collecting soil samples from a discrete or isolated subsurface interval, without the need of extracting excess material above the target depth. These methods dramatically reduce time and cost associated with disposal of material from soil cuttings when compared to test pit sampling. In addition, direct-push and split-spoon sampling methods can obtain samples at targeted intervals greater than 15 feet in depth, allowing for discrete depth soil sampling while speeding up the sampling process. Direct-push methods work best in medium to fine-grained cohesive materials such as medium to fine sands, silts, and silty clay soils. Split-spoon sampling works well in all types of soil, but is somewhat slower than direct-push methods. Samples are composited so that each sample contains a homogenized representative portion of the sample interval. Due to potential loss of analytes, samples for volatile analysis are not composited. Samples for chemical analysis can be collected by any of the above-mentioned sampling methods, as disturbed soil samples. Undisturbed samples are collected, sealed, and sent directly to the laboratory for analysis. For undisturbed samples, the samples are not homogenized.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Delivery Order (DO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.

2.2 Before soil sampling commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated soil sampling locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Low recovery of soil from sampling equipment will prevent an adequate representation of the soil profile and sufficient amount of soil sample. If low recovery is a problem, the hole may be offset and re-advanced, terminated, or continued using a larger diameter sampler.

4.2 Asphalt in soil samples can cause false positive results for hydrocarbons. To ensure samples are free of asphalt, do not collect samples that may contain asphalt. If the collection of samples potentially containing asphalt is unavoidable, note the sampling depths at which the presence of asphalt are suspected.

4.3 Instrumentation interferences addressed in SOPs for Calibration of the Photoionization Detector (PID), Headspace Screening for Total Volatile Organics, and Equipment Decontamination must also be considered.

4.4 Cross contamination from sampling equipment must be prevented by using sampling equipment constructed of stainless steel that is adequately decontaminated between samples.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The DO Manager is responsible for ensuring that soil sampling activities comply with this procedure. The DO Manager is responsible for ensuring that all personnel involved in soil sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all soil sampling activities are conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

The depth at which samples will be collected and the anticipated method of sample collection (direct-push, split-spoon, hand auger, shovel, or test pits) will be presented in the SAP. The following details equipment typically needed for soil sampling, based on the various methods. See the SAP for specific detail of equipment and supply needs.

6.1 Depending on the nature of suspected contamination, field screening instrumentation may be used for direct sampling. Appropriate instrumentation and calibration standards should be available. If volatile organic contaminants are suspected and a PID will be used, refer to the equipment and instrumentation listed in SOP 3-20 Operation and Calibration of a Photoionization Detector. Equipment in this SOP includes but is not limited to:

- PID/FID;
- Calibration gas; and
- Tedlar® gas bags (for calibration).

6.2 If field screening methods include jar or zipper plastic bag headspace screening for volatile organics, refer to the equipment and procedure in SOP 3-19 Headspace Screening for Total VOCs. Equipment in this SOP includes but is not limited to:

- Zipper lock plastic bags, or
- Clean soil (“drillers jars”) jars; and
- Aluminium foil.

6.3 Appropriate decontamination procedures must be followed for sampling equipment. Refer to SOP 3-06 Equipment Decontamination. Equipment in this SOP includes but is not limited to:

- Phosphate-free detergent;
- Isopropyl Alcohol;
- Tap water;
- Deionized Ultra-Filtered (DIUF) Water;
- Plastic buckets or washbasins;
- Brushes; and

- Polyethylene sheeting.

6.4 The following general equipment is needed for all soil sampling, regardless of method:

- Stainless steel bowls;
- Stainless steel trowels;
- Appropriate sample containers for laboratory analysis;
- Personal Protective Equipment (PPE);
- Logbook;
- Cooler and ice for preservation; and
- Stakes and flagging to document sampling location.

6.5 The following additional equipment is needed for volatile organic sampling:

- Electronic pan scale and weights for calibration; and
- Syringes or other discrete soil core samplers (i.e., TerraCore® or EnCore®).

6.6 The following additional equipment may be needed for surface and test pit soil sampling:

- Hand Auger

6.7 The following additional equipment may be needed for soil sampling from direct push and/or split-spoon equipment:

- Tape measure or folding carpenter's rule for recording the length of soil recovered.

Note: All subsurface drilling equipment will be provided and maintained by the subcontractor.

7.0 Procedure

7.1 **General Soil Sampling Procedure for All Soil Sampling Methods**

7.1.1 Record the weather conditions and other relevant on-site conditions.

7.1.2 Select the soil sampling location, clear vegetation if necessary, and record the sampling location identification number and pertinent location details.

7.1.3 Verify that the sampling equipment is properly decontaminated, in working order, and situated at the intended sampling location.

7.1.4 Place polyethylene sheeting on the ground and assemble all necessary sampling equipment on top of it. Cover surfaces onto which soils or sampling equipment will be placed (i.e. tables with polyethylene sheeting).

7.1.5 Follow the appropriate procedures listed below for either surface, split-spoon, direct push, or test pit sample collection (7.2, 7.3, 7.4, and 7.5 respectively).

7.1.6 Collect soil samples according to procedures listed in Section 7.6 depending on project specific analyses.

7.1.7 Record date/time, sample ID, and sample descriptions in the field logbook or field form. A sketch or description of the location may also be recorded so the sample location can be re-constructed, especially if the location will not be recorded using global positioning satellite (GPS) equipment.

7.1.8 Immediately label the sample containers and place them on ice, if required for preservation. Complete the chain-of-custody form(s) as soon as possible.

7.1.9 Dispose of all excess excavated soil in accordance with the SAP.

7.1.10 If required, mark the sample location with a clearly labelled wooden stake or pin flag. If the location is on a paved surface, the location may be marked with spray paint.

7.1.11 Decontaminate the sampling equipment according to SOP 3-06 Equipment Decontamination.

7.2 **Surface Sampling**

7.2.1 The criteria used for selecting surface soil locations for sampling may include the following:

- Visual observations (soil staining, fill materials);
- Other relevant soil characteristics;
- Site features;
- Screening results;
- Predetermined sampling approach (i.e. grid or random); and
- Sampling objectives as provided in the SAP.

7.2.2 The following procedures are to be used to collect surface soil samples. Surface soils are considered to be soils that are up to two feet below ground surface, though state regulations and project objectives may define surface soils differently; therefore, the SAP should be consulted for direction on the depth from which to collect the surface soil samples. Sampling and other pertinent data and information will be recorded in the field logbook and/or on field forms. Photographs may be taken as needed or as specified in the SAP.

1. Gently scrape any vegetative covering until soil is exposed. Completely remove any pavement.
2. Remove soil from the exposed sampling area with a trowel, hand auger, or shovel. Put soils within the sampling interval in a stainless steel bowl for homogenizing. Monitor the breathing zone and sampling area as required in the HASP.
3. For VOC analyses, collect representative soil samples directly from the recently-exposed soil using a syringe or other soil coring device (e.g., TerraCore®, EnCore®). Follow procedures in Section 7.6.1 for VOC sampling.
4. Collect sufficient soil to fill all remaining sample jars into a stainless steel bowl. Homogenize the soil samples to obtain a uniform soil composition which is representative of the total soil sample collected according to the following procedure:
    a) Remove all rocks and non-soil objects using a stainless steel spoon or scoop.
    b) Form a cone shaped mound with the sample material, then flatten the cone and split the sample into quarters.
    c) Use the stainless steel spoon/scoop to mix the quarter samples that are opposite.
    d) After mixing the opposite quarters, reform the cone shaped mound.
    e) Repeat this procedure a minimum of five (5) times, removing any non-soil objects and breaking apart any clumps.

7.3 **Split-Spoon Sampling**

7.3.1 At each boring location, the frequency and depth of split-spoon samples will be determined from the SAP. Split-spoon samples may be collected continuously, intermittently, or from predetermined depths.

7.3.2 Split-spoon samplers shall be driven into undisturbed soil by driving the spoon ahead of the drill augers/casing. In cohesive soils, or soils where the borehole remains open (does not collapse), two split-spoon samples may be taken prior to advancing the augers/casing.

7.3.3 After split-spoons are retrieved, open the split-spoon and measure the recovery of soil. If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the

depth at which it was observed along with all other pertinent observations. If required in the SAP, VOC and headspace samples should be collected (see Section 7.6.1) prior to logging the sample.

7.3.4 If headspace screening for VOCs is required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.3.5 Soils collected using the split-spoon sampler will be logged by the field representative using the procedure required in the SAP.

7.3.6 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.3.7 The SAP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from that interval will be set aside in a clearly labelled stainless steel bowl covered with aluminium foil. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.

7.3.8 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

7.4 Direct Push Sampling

At each boring location, the frequency of direct-push samples will be determined from the SAP. Typically, samples with direct-push equipment are collected in 4 foot (ft) intervals, but smaller (e.g., 2 ft) and larger (e.g., 5 ft) intervals are also possible.

1. Sample using Macro-Core samplers with acetate liners to obtain discrete soil samples at the depths specified in the SAP.
2. Cut open the acetate liner. If required in the SAP, immediately scan the recovered soil boring for VOCs using a PID by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. VOC and headspace samples, if required in the SAP should be collected (see Section 7.6.1) prior to logging the sample.
3. If required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.
4. Soils collected using the direct-push sampler will be logged by the field by the field representative using the procedure required in the SAP.
5. Collect the remainder of the sample into a stainless steel bowl. Homogenize the soil collected so that the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.
6. Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

7.5 Test Pit Sampling

7.5.1 Excavate the test pit to the desired depth.

7.5.2 Using the excavator bucket, collect soil samples as specified in the SAP. Collect a sample and perform screening analyses as required by the SAP. If VOCs contamination is suspected, perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.5.3 Collect the sample from the center of the bucket to avoid potential contamination from the bucket.

7.5.4 VOC samples should also be collected from an undisturbed section soil in the excavator bucket. The top layer of exposed soil should be scraped away just prior to collecting the VOC samples.

7.5.5 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.5.6 Dispose of all excavated soil according to the SAP.

7.6 **Sample Collection Methods**

7.6.1 Volatile Organics Sampling

For soils collected for analyses of volatile organics, including Volatile Petroleum Hydrocarbons (VPH) or other purgable compounds, a closed system is maintained. From collection through analysis, the sample bottles are not opened. The bottle kit for a routine field sample for these analyses will typically include three 40-mL VOA vials and one soil jar. Two 40-mL VOA vials will contain either 5 mL reagent water or 5 mL sodium bisulfate and magnetic stir bars (i.e., low level vials). The third VOA vial will contain 15 mL methanol with no magnetic stir bar (i.e., high level vial). These vials are usually provided by the laboratory and are pre-weighed, with the tare weight recorded on the affixed sample label. No additional sample labels are affixed to the VOA vials, as addition of a label would alter the vial weight. All information is recorded directly on the sample label using an indelible marker. The soil jar is provided for percent solids determination. For VOC or VPH analyses, samples are collected prior to sample homogenization. Collect the VOC sample in accordance with the procedure described below.

1. Determine the soil volume necessary for the required sample weight, typically 5 grams:
   a) Prepare a 5 mL sampling corer (e.g., Terra Core®) or cut-off plastic syringe.
   b) Tare the sampler by placing it on the scale, and zeroing the scale.
   c) Draw back the plunger to the 5 gram mark or 5mL (5cc) mark on cut-off syringe, and insert the open end of the sampler into an undisturbed area of soil with a twisting motion, filling the sampler with soil. Note the location of the plunger with respect to the milliliter (cc) or other graduation printed on the sampler.
   d) Weigh the filled sampler, and remove or add soil until the desired weight is obtained. Note the location of the plunger which corresponds to this weight. Do not use this sample for laboratory analysis.
2. Once the required soil volume has been determined, pull the plunger back to this mark and hold it there while filling the syringe for each sample.
3. Collect 5 grams of soil using the cut-off syringe or Terra Core® sample device. Extrude the 5-grams of soil into one of the low level 40-mL VOA vials. Quickly wipe any soil from the threads of the VOA vial with a clean Kimwipe® and immediately close the vial. It is imperative that the threads be free from soil or other debris prior to replacing the cap on the vial in order to maintain the closed system necessary for the analysis.
4. Gently swirl the vial so that all of the soil is fully wetted with the preservative.
5. Fill the other low level 40 mL VOA vial in this manner.
6. Repeat the process for the high level VOA vials, only for the high level VOA vial three 5 gram aliquots (i.e., 15 grams total) should be extruded into the high level VOA vial.

   NOTE: Depending on the laboratory, some high level VOA vials only contain 5 mL or 10 mL of methanol. If this is the case, either 5 grams total or 10 grams total, respectively, should be extruded into the high level VOA vial. In other words, the mass of soil in grams should be identical to the volume of methanol in mL (i.e., 1:1 ratio of soil to methanol).
7. Collect any additional QC sample collected (e.g., field duplicate, MS, and MSD) in the same manner as above.
8. Fill the 2-oz or 4-oz glass jar with soil from the same area for percent moisture determination.

7.6.2 Soil Sampling Method (All other analyses except VOC/VPH)

When all the required soil for a sampling location has been obtained, the soil can be homogenized as described in section 7.2. Collect sufficient volume to fill all of the remaining sample containers at least ¾ full for all other analyses. Homogenize the soil in a decontaminated stainless steel bowl, removing rocks,

sticks, or other non-soil objects and breaking apart any lumps of soil prior to filling the remaining sample containers.

NOTE: Soil samples must contain greater than 30% solids for the data to be considered valid.

8.0 Quality Control and Assurance

8.1 Sampling personnel should follow specific quality assurance guidelines as outlined in the SAP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the SAP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

8.2 Quality control requirements are dependent on project-specific sampling objectives. The SAP will provide requirements for equipment decontamination (frequency and materials), sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

9.0 Records, Data Analysis, Calculations

All data and information (e.g., sample collection method used) must be documented on field data sheets, boring logs, or within site logbooks with permanent ink. Data recorded may include the following:

- Weather conditions;
- Arrival and departure time of persons on site;
- Instrument type, lamp (PID), make, model and serial number;
- Calibration gas used;
- Date, time and results of instrument calibration and calibration checks;
- Sampling date and time;
- Sampling location;
- Samples collected;
- Sampling depth and soil type;
- Deviations from the procedure as written; and
- Readings obtained.

10.0 Attachments or References

SOP 3-06, *Equipment Decontamination*

SOP 3-19, *Headspace Screening for Total VOCs*

SOP 3-20, *Operation and Calibration of a Photoionization Detector*

Sediment Sampling

Procedure 3-22

1.0 Purpose and Scope

1.1 Sediment contamination is a widespread environmental problem that can pose a threat to a variety of aquatic ecosystems. Sediment functions as a reservoir for common contaminants such as pesticides, herbicides, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and metals such as lead, mercury, and arsenic. Contaminated sediments represent a hazard to aquatic life through direct toxicity, as well as to aquatic life, wildlife, and human health through bioaccumulation. Accurate assessment of environmental hazards posed by sediment contamination depends in large part on the accuracy and representativeness of sediment collection and analyses (U.S. EPA, 2001).

1.2 Selection and proper use of sediment sampling equipment is essential to the collection of accurate, representative sediment data that will meet the project Data Quality Objectives (DQOs). Most sediment collection devices are designed to isolate and consistently retrieve a specified volume and surface area of sediment, from a required depth below the sediment surface, with minimal disruption of the integrity of the sample and no contamination of the sample. Maintaining the integrity of the collected sediment, for the purposes of the measurements intended, is a primary concern in most studies because disruption of the sediment's structure could change its physiochemical and biological characteristics, thereby influencing the bioavailability of contaminants and the potential toxicity of the sediment (U.S. EPA, 2001).

When selecting the type of sediment sampling equipment to be used for an event, the project DQOs as well as the sediment characteristics should be considered. Related to the project DQOs is the desired depth of sediment sampling. For monitoring and assessment studies where historical contamination is not the focus, the upper 10 to 15 centimeters (cm) is typically the horizon of interest, as this is the horizon that generally contains the most recently deposited sediments and most epifaunal and infaunal organisms (U.S. EPA, 2001). The 0-6 inches interval for sediments with less than two feet of water is also used for human health risk assessment purposes. Sampling of these horizons can usually be done with grab samplers. However, if sediment contamination is being related to organism exposures (e.g. benthic macroinvertebrates and/or fish), or if characterization of deeper sediments is important for comparison of recent surficial versus historical contamination, then more precise sampling of sediment depths might be needed, and a hand corer may be more suitable (U.S. EPA, 2001).

1.3 This standard operating procedure (SOP) describes the procedure for the collection of sediment samples using the Petite Ponar® Grab Sampler, Ekman Bottom Grab Sampler, and Wildco® Hand Corer (or similar sampling devices). The applicability of each of the sediment samplers is described below.

The Petite Ponar® Grab Sampler is used to collect sediment samples in:

- Firm, hard bottoms such as sand, gravel, consolidated marl, and clay
- Mixtures of sand, stones, and coarse debris
- Soft or mucky sediments

The Ekman Bottom Grab Sampler is used to collect sediment samples in:

- Soft, finely divided littoral bottoms free from vegetation and intermixtures of sand, stones, and other coarse debris
- Bottoms composed of finely divided mulch, mud, muck, or submerged fine peaty materials

The Wildco® Hand Corer is used:

- To collect sediment samples for geological characterizations and dating
- To collect sediment samples for programs where it is important to maintain an oxygen-free environment for the sample during collection
- To collect sediment samples from a deeper depth than a grab sampler, and to characterize the depth of contamination at a site

- To investigate the historical input of contaminants to aquatic systems
- To collect sediment samples in semi-consolidated and soft sediment

Pictures and exploded diagrams of the Petite Ponar Grab Sampler, Ekman Bottom Grab Sampler, and Wildco® Hand Corer are presented in Figures 1, 2, and 3, respectively.

1.4 This procedure is the Program-approved professional guidance for work performed by AECOM-Tidewater, Inc. Joint Venture (AECOM-Tidewater JV).

1.5 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first location. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring of sample locations to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and solid or liquid matrix through the use of respirators and disposable clothing.

2.2 Observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during sediment sampling activities includes inner disposable vinyl or nitrile gloves, outer chemical-protective nitrile gloves, and waders (if applicable). Refer to the project-specific HASP for the required PPE.

2.3 Handle all sediments removed from potentially contaminated locations in accordance with the IDW handling procedures in SOP 3-05, IDW Management.

2.4 Depending upon the type of contaminant expected or determined in previous sampling efforts, employ the following safe work practices:

- If sampling from a boat, all sampling personnel should wear personal flotation devices (PFDs) when in the boat, and should follow all health and safety protocols for working in a boat presented in the project-specific HASP.
- Lifting the samplers into the boat, dumping its contents, and washing those contents may require leaning over the side of the boat. Care should be taken to keep the boat in proper balance at all times during sampling.
- Severe injury to fingers or hands can be caused by movement of the lever arms of the Petite Ponar® Grab Sampler. Do not handle or move the Petite Ponar® Grab Sampler unless the safety pin is fully inserted in the locking holes.
- Severe injury to fingers or hands can be caused by the closing of the sharpened scoops of the Ekman Bottom Grab Sampler. Handle the Ekman Bottom Grab Sampler very carefully when the springs are set and the cable loops are hooked (armed) on the Twin-Pin™ pins on the release mechanism. Do not “arm” the Ekman Bottom Grab Sampler until the sampler is ready to be used. The Ekman Bottom Grab Sampler spring-loaded jaws are potentially dangerous; extreme care must be exercised when setting the jaws. To prevent injury (and to extend the life of the springs), unhook both springs from their scoop buttons after each sampling session.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Delivery Order (DO) Manager** is responsible for ensuring that sediment sampling activities comply with this procedure. The **DO Manager** is responsible for ensuring that all field sampling personnel involved in sediment sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all field sampling personnel follow these procedures.

4.4 **Field sampling personnel** are responsible for the implementation of this procedure.

4.5 The field sampler and/or task manager is responsible for directly supervising the sediment sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling.

5.0 Equipment and Supplies

5.1 For sediment sampling using all types of equipment, the following supplies are required:

- Stainless steel bowls
- Stainless steel hand trowels, spoons, spatulas, and scoops
- Munsell Color Chart
- Particle size chart

5.2 Petite Ponar® Grab Sampler

- 3/16" braided polyester line
- Auxiliary weights

5.3 Ekman Bottom Grab Sampler

- 11 oz split messenger
- 3/16" braided polyester line
- Extension Handle
- Auxiliary weights

5.4 Wildco® Hand Corer

- 3/16" braided polyester line
- Extension handle
- Stainless steel core catchers (for normal sediments)
- Eggshell™ core catchers (for wet sediments)
- Stainless steel nose piece
- Cellulose acetate butyrate (CAB) liners
- Core liner end caps
- Core liner cutter
- Geologists table
- Auxiliary weights

6.0 Procedure

6.1 Depending on the characteristics of the site being investigated, sediment samples may be collected from a boat, or by sampling personnel in waders. In all instances, sediment sampling should begin from the most downstream location and proceed to the most upstream location. If sediment samples are collocated with surface water samples, the surface water sample should be collected prior to the sediment sample in order to avoid increased turbidity from displaced sediment. Regardless of the type of sediment sampling equipment used, documentation of field observations and collection activities should be recorded on the sediment sampling sheet or electronic data collection device. The following

observations should be recorded on the sediment sampling form (see Attachment 1) for all sediment sampling activities:

- Sample location
- Weather conditions and other relevant site conditions
- Depth of water to the nearest 0.1 foot. A surveyor rod may be used. If the surveyor rod is used, minimize water turbulence and do not disturb any sediment.
- Physical characteristics of the water body such as estimated current speed (stagnant, slow, medium, or fast) and direction, odor, color, presence of any dead vegetation, surface sheens, etc.
- Sediment color according to the Munsell Color Chart
- Sediment grain size according to a particle size chart

Specific procedures for the collection of sediment samples using the Petite Ponar® Grab Sampler, Ekman Bottom Grab Sampler, and Wildco® Hand Corer are presented below.

6.2 Petite Ponar® Grab Sampler

6.2.1 Inspect the sampler to ensure all parts are in good working condition.

6.2.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.2.3 Attach the 3/16" braided polyester line to the sampler by looping the line through the clevis at the top center of the lever arms and tying securely. Tie the other end of the line to the boat (if applicable), or make sure to hold on to the other end of the line. Strong, tight knots (e.g. bowline, two half hitches) are essential for operator safety and to prevent losing the sampler. If necessary, attach the auxiliary weights to the sampler according the manufacturer's directions.

6.2.4 Insert the Pinch-Pin™ into its hole in the lever arms, making sure to firmly push the Pinch-Pin™ into the hole. As long as the line is taut, the Pinch-Pin™ will stay in its place. When the line becomes the least bit slack (e.g. when the sampler hits the bottom), the Pinch-Pin™ spring will force the Pinch-Pin™ out of its hole, allowing the scoops to close.

6.2.5 Just before lowering the grab into the water, and with the line taut, remove the safety pin so the closing mechanism will release when the sampler is on the bottom. Make sure to keep the line taut, as any loss of tension in the line will cause the Pinch-Pin™ to pop out, closing the sampler.

6.2.6 Lower the sampler into the water in a slow and controlled fashion, especially during the final 1-2', such that the bow wave is minimized, thus minimizing the dispersal of fine material on the sediment surface. At no time should the sampler be allowed to "free fall" down through the water column.

6.2.7 Once the sampler has reached the bottom, release the tension on the line, and allow the sampler to sink into the sediment momentarily. The release of tension on the line will cause the Pinch-Pin™ to pop out.

6.2.8 Collect the sample by pulling on the line, which will cause the lever arms to drive the scoops into the sediment in a closing motion. Keep pulling on the line in a controlled fashion until the scoops drive through the sediment and close.

6.2.9 Once the sampler scoops have closed, continue pulling on the line in a controlled fashion in order to retrieve the sampler back to the surface. When the sampler reaches the surface, lift it clear and bring it above a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.

6.2.10 Prior to sampling and sample homogenization, the overlying water in the sampler should be siphoned off, and not decanted (U.S. EPA 2001).

6.2.11 If acid volatile sulfide/simultaneously extracted metals (AVS/SEM) samples are to be collected, open the top screens of the sampler and collect the AVS/SEM sample directly from the sediment contained in the sampler according to the procedures specified in the project-specific SAP.

6.2.12 If volatile organic compound (VOC) samples are to be collected, open the top screens of the sampler and collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the undisturbed sediment contained within the sampler, making sure to follow all VOC sampling procedures specified in the project-specific SAP. Once the VOC samples have been collected,

collect an additional aliquot for the VOC percent solids sample directly from the undisturbed sediment contained within the sampler.

6.2.13 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), open the sampler by pulling the two scoops open, taking care to keep hands and fingers away from the sharpened edges of the scoops, and allow the sediment to exit the sampler into the decontaminated stainless steel bowl.

6.2.14 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.2.3 through 6.2.12 until an adequate sample volume has been collected, taking care to deploy the sampler to an area that is proximal and upstream, but not on top of, the previous sample location.

6.2.15 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).

6.3 Ekman Bottom Grab Sampler with the 11 oz Split Messenger

6.3.1 Inspect the sampler to ensure all parts are in good working condition.

6.3.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.3.3 Attach the 3/16" braided polyester line to the sampler by passing the line through the trip mechanism and knotting it securely below the underlying plate. Thread the 11 oz split messenger on the line, and tie the other end of the line to the boat (if applicable), or make sure to hold on to the other end of the line. Strong, tight knots (e.g. bowline, two half hitches) are essential to prevent losing the sampler. If necessary, attach the auxiliary weights to the sampler according the manufacturer's directions.

6.3.4 Set the spring on the side of the sampler by hooking the end of the spring onto one scoop button and stretching the spring to reach the second scoop button. Repeat this procedure with the spring on the other side of the sampler.

6.3.5 Arm the scoops by hooking one cable loop to one Twin-Pin™ pin in the trip assembly on the top of the sampler. The white ball on the cable can be used as a hand grip to assist getting the cable loop hooked onto the Twin-Pin™ pin. Repeat for the opposite cable loop. The sampler is now armed and dangerous. Do not allow anything to come in contact with the trip assembly at the top of the sampler, as this may cause a sudden and unexpected closure of the sampler.

6.3.6 Lower the sampler into the water in a slow and controlled fashion, especially during the final 1-2', such that the bow wave is minimized, thus minimizing the dispersal of fine material on the sediment surface. At no time should the sampler be allowed to "free fall" down through the water column.

6.3.7 Once the sampler has reached the bottom, allow the sampler to settle momentarily. Once the sampler has settled, hold the line with just enough tension to keep it straight, and send the 11 oz split messenger down the line. Once the 11 oz split messenger impacts Twin-Pin™ strike pad in the trip assembly on the top of the sampler, the two cable loops will be released from the Twin-Pin™ pins, and the spring-loaded scoops of the sampler will automatically close.

6.3.8 Retrieve the sampler by pulling up the line in with a moderate, steady speed. When the sampler reaches the surface, lift it clear and bring it above a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.

6.3.9 Prior to sampling and sample homogenization, the overlying water in the sampler should be siphoned off, and not decanted (U.S. EPA 2001).

6.3.10 If AVS/SEM samples are to be collected, open the top lids of the sampler and collect the AVS/SEM sample directly from the sediment contained in the sampler according to the procedures specified in the project-specific SAP.

6.3.11 If VOC samples are to be collected, open the top lids of the sampler and collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the undisturbed sediment contained within the sampler, making sure to follow all VOC sampling procedures specified in the project-specific SAP. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the undisturbed sediment contained within the sampler.

6.3.12 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), open the sampler by pulling on the white balls on both cables, opening the spring-loaded scoops and allowing the sediment to exit the sampler into the decontaminated stainless steel bowl. While the spring-loaded scoops are being held open, do not place hands or fingers inside or underneath the sampler.

6.3.13 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.3.4 through 6.3.11 until an adequate sample volume has been collected, taking care to deploy the sampler to an area that is proximal and upstream, but not on top of, the previous sample location.

6.3.14 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).

6.4 Ekman Bottom Grab Sampler with the Extension Handle

6.4.1 Inspect the sampler to ensure all parts are in good working condition.

6.4.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.4.3 Attach the extension handle to the top of the sampler with machine bolts.

6.4.4 Arm the sampler according to the procedures described in steps 6.3.3 and 6.3.4 above.

6.4.5 Using the extension handle, lower the sampler to a point 4-6" above the sediment surface, and drop the sampler to the sediment, keeping the sampler vertical at all times.

6.4.6 Trigger the trip assembly by depressing the button on the upper end of the extension handle. This will cause the two cable loops to be released from the Twin-Pin™ pins, and the spring-loaded scoops of the sampler will automatically close.

6.4.7 While keeping the sampler vertical, bring the sampler over to a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.

6.4.8 Collect samples according to the procedures described in steps 6.3.8 through 6.3.13 above.

6.5 Wildco® Hand Corer with the Push Handles

6.5.1 Inspect the sampler to ensure all parts are in good working condition:

- Assemble and disassemble the core tube from the head and nose piece to make sure the threads are not binding. If the threads are binding, consult the manufacturer's directions.
- Make sure that the CAB plastic liner can slide easily in and out of the core tube.
- Make sure the bottom edge of the core tube and nose piece are sharp and free from nicks or dents. If necessary, file smooth using a round file.
- Check the flutter valve for ease of movement.
- Check the flutter valve seat to make sure it is clear of any obstruction, disfigurement, grease, and/or oil that could prevent a tight closure.

6.5.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.5.3 Screw the corer head onto the core tube, and screw the two handles onto the corer head.

6.5.4 Insert a CAB plastic liner into the core tube, insert a core catcher onto the end of the CAB plastic liner (stainless steel for normal sediments, Eggshell™ for wet sediments), and screw the stainless steel nose piece onto the core tube. If using the hand corer from a boat, bridge, high dock, etc., be sure that the appropriate extension handle (5', 10' or 15') is attached to the corer head.

6.5.5 Get in position over the sampling location. If wading in shallow water, be sure to approach the sample location from the downstream side. Line up the sampler, aiming it vertically for the point where the sample is being taken, and push the hand corer in a smooth continuous motion through the water and into the sediment. Increase the thrust as necessary to obtain the penetration desired. Do not hammer or pound the corer into the sediment.

6.5.6 Retrieve the sample by pulling straight up on the handles, keeping the corer as vertical as possible. If the corer has not been completely submerged, close the flutter valve by hand and press it shut while the sample is being retrieved. The flutter valve must be kept very wet if it is to seal properly and prevent sample washout. If the substrate is gripping the corer too tightly, gently rock the top of the corer back and forth horizontally to increase the size of the hole created by the corer and reduce the pull-out suction.

6.5.7 Unscrew the nose piece from the corer and cap the bottom end of the CAB core liner. Release the flutter valve to free the CAB core liner, and slide the CAB core liner from the core tube. Cap the top of the CAB core liner and inspect the CAB core liner for recovery. If the recovery is adequate, proceed to step 6.5.8. If the recovery is not adequate, resample the location by repeating steps 6.5.3 through 6.5.7.

6.5.8 Bring the CAB core liner with the sediment sample over to the geologist table, keeping the core vertical. Place the CAB core liner on the geologist table and cut open with a core liner cutter. If AVS/SEM samples are to be collected, collect the AVS/SEM sample directly from the sediment contained in the core liner according to the procedures specified in the project-specific SAP. If VOC samples are to be collected, collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the sediment core. Consult the project-specific SAP for project-specific VOC sediment sampling procedures. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the sediment core.

6.5.9 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), use a decontaminated stainless steel spoon to transfer the remaining sediment core into a decontaminated stainless steel bowl.

6.5.10 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.5.3 through 6.5.8 until an adequate sample volume has been collected, taking care to deploy the corer to an area that is proximal, but not on top of, the previous sample location.

6.5.11 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).

6.6 Wildco® Hand Corer with the Clevis and Line

6.6.1 Inspect the corer as described in step 6.5.1 above.

6.6.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.

6.6.3 Screw the corer head onto the core tube. Attach the 3/16" braided polyester line to the corer by passing the line through the clevis in the corer head and knotting it securely. Strong, tight knots are essential to prevent losing the corer. If necessary, attach the auxiliary weights to the sampler according the manufacturer's directions.

6.6.4 Insert a CAB plastic liner into the core tube, insert a core catcher onto the end of the CAB plastic liner (stainless steel for normal sediments, Eggshell™ for soupy sediments), and screw the stainless steel nose piece onto the core tube.

6.6.5 Position the corer over the drop point and steady momentarily, making sure to keep the corer vertical at all times. Make sure to arrange the 3/16" braided polyester line to run freely. Since the corer's penetration is by simple gravity, it is important that there be no restraint on the corer during descent by stricture on the line. Keep a firm hold on the free end of the line, or tie it to the boat (if applicable) or some other permanent fixture.

6.6.6 Drop the corer into the water, and allow the corer to free fall until it hits the sediment surface. The corer should not be dropped to depths greater than 20' to 30'. Dropping the corer to depths greater than 20' to 30' may result in the corer striking the sediment surface at an angle less than 90°, resulting in an unsatisfactory sample.

6.6.7 Once the corer has entered the sediment and is no longer falling, draw the line taut, and then pull on the line to pull the corer from the sediment. Once the corer has been pulled free from the sediment, bring the corer back to the surface by pulling up the line, using a smooth, hand-over-hand fashion. This movement automatically causes the flutter valve to close, preventing sample washout in all but the soupiest of sediments.

6.6.8 Once the corer has been returned to the surface, lift the corer clear of the water, being careful to keep the corer as vertical as possible at all times.

6.6.9 Collect the sediment sample according to the procedures outlined in steps 6.5.6 through 6.5.11 above.

7.0 Quality Control and Assurance

7.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

7.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

8.0 Records, Data Analysis, Calculations

8.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Sample Collection Records;
- Field logbook;
- Chain-of-custody forms; and
- Shipping labels.

8.2 Sample collection records (Attachment 1) will provide descriptive information for the sediment samples collected at each location.

8.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.

8.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

8.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

9.0 Attachments or References

Attachment 1 – Sediment Sample Collection Record

Figure 1 – Petite Ponar® Grab Sampler and Exploded Diagram

Figure 2 – Ekman Bottom Grab Sampler (Large, Tall, and Standard Sizes) and Exploded Diagram

Figure 3 – Wildco® Hand Corer (with Case and Accessories) and Exploded Diagram

Figure 4 – Illustrations of Acceptable and Unacceptable Grab Samples

DoD Environmental Field Sampling Handbook, Revision 1.0. April 2013.

U.S. Environmental Protection Agency (U.S. EPA). 2001. *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual.* October.

Wildlife Supply Company. 2003. 2424- Hand Corer Instructions.

Wildlife Supply Company. 2004. Ekman Bottom Grabs Instructions and Maintenance.

Wildlife Supply Company. 2004. 1728-G30/ 1728-G40 Petite Ponar® Grab.

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

Attachment 1
Sediment Sample Collection Record

AECOM Any Task – Anywhere TIDEWATER INC

SEDIMENT SAMPLE COLLECTION FORM

Project Name:
Date(s):

Project #:		**Date:**	
Sample Location ID:		**Time:**	
Sample #:		**Weather:**	
Samplers:			

Sample Information:

Sample Depth:		**Sampling Device:**	
Water Depth:			
Distance from River Bank:			
River Flow Rate:			
Field Decon:	Yes No Dedicated	**Sample Type:**	Grab Composite

Munsell Color:

Sample Description:

Other physical characteristics of water body at sample location:
(Water color, turbidity, odor, presence of sheens, dead/stressed vegetation, etc.)

Sample Comments/Description:

Figure 1
Petite Ponar® Grab Sampler and Exploded Diagram

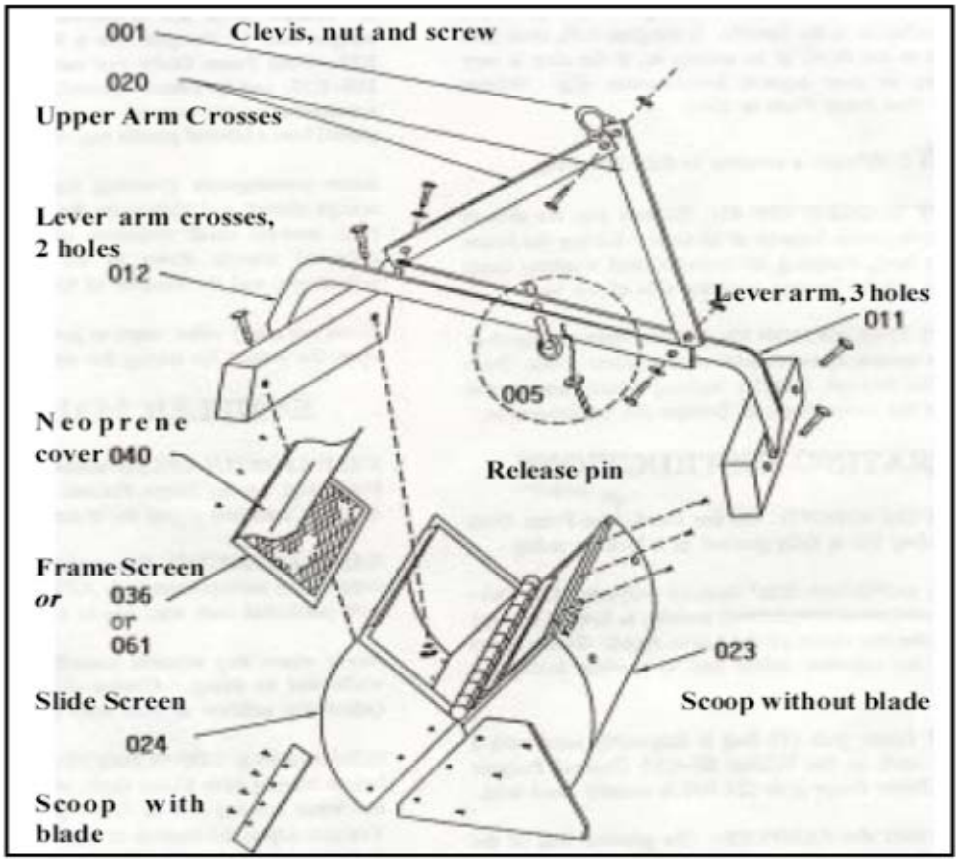
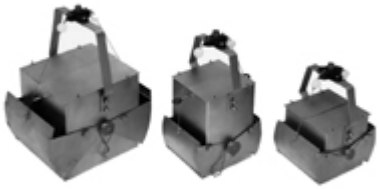


Figure 2

Ekman Bottom Grab Sampler (Large, Tall, and Standard Sizes) and Exploded Diagram



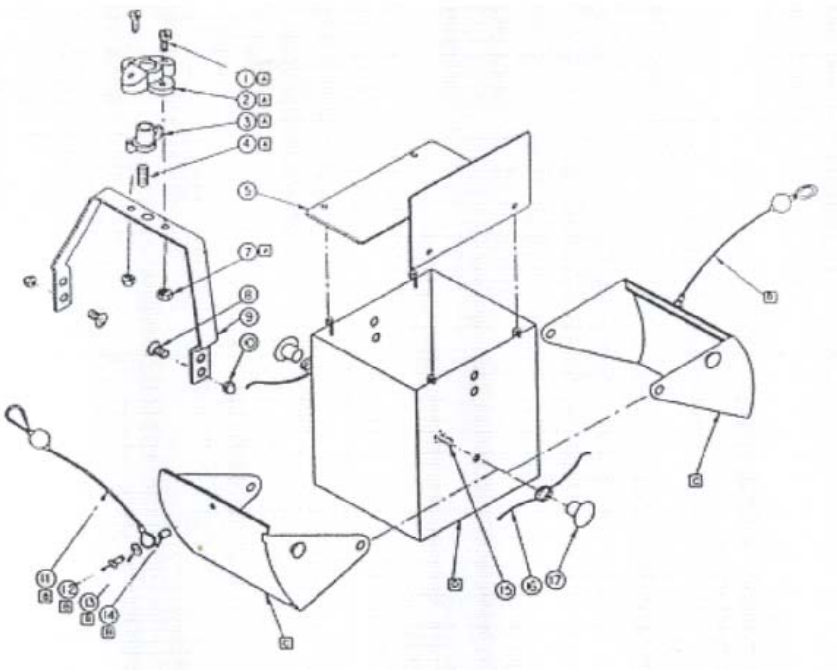
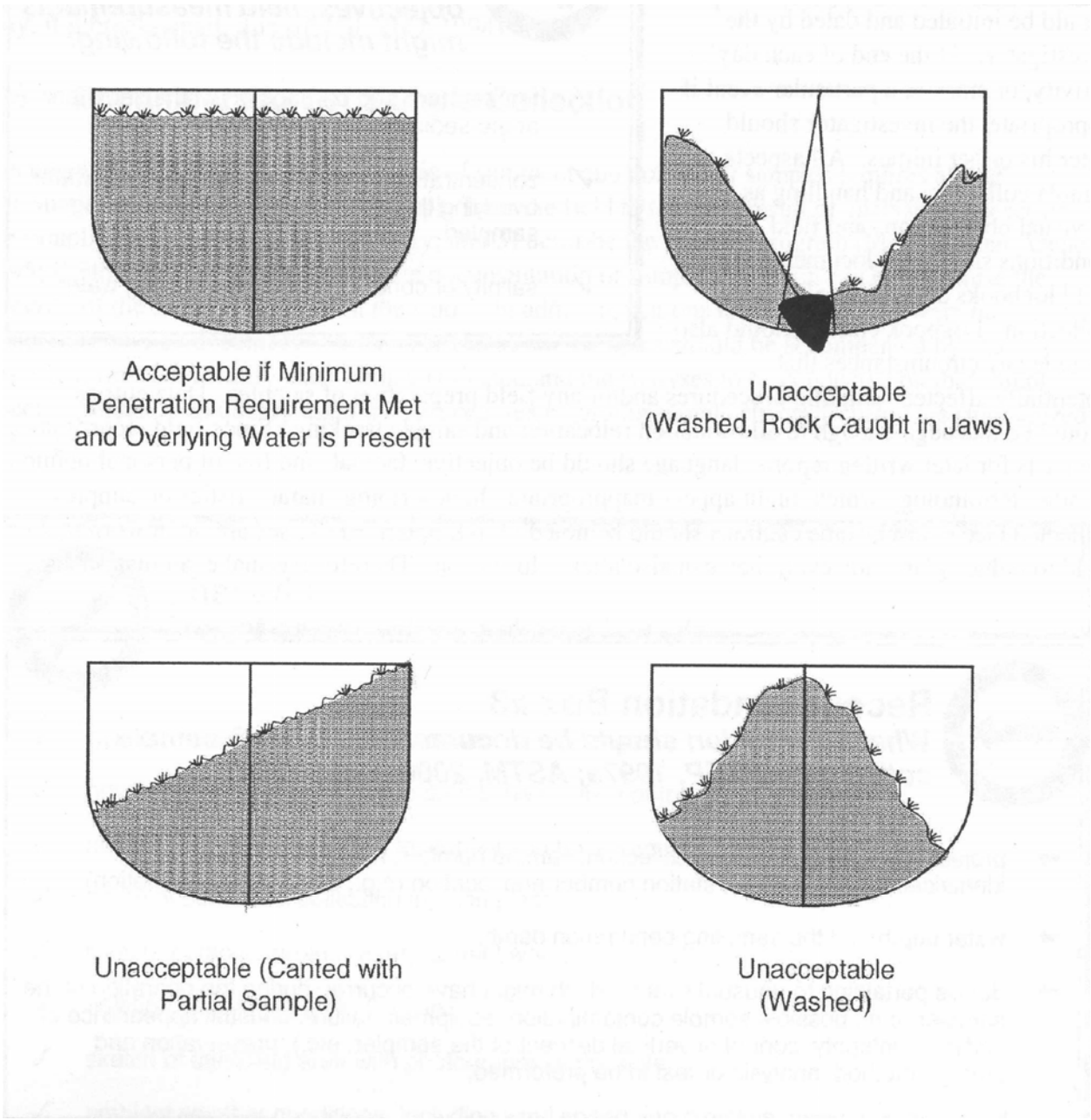


Figure 3
Wildco® Hand Corer (with Case and Accessories) and Exploded Diagram

Figure 4
Illustrations of Acceptable and Unacceptable Grab Samples



Landfill Gas Screening Procedures

1.0 Scope and Application

This is a Standard Operating Procedure (SOP) for landfill gas (LFG) screening for methane (CH_4), carbon dioxide (CO_2), carbon monoxide (CO), oxygen (O_2), and hydrogen sulfide (H_2S) to achieve the following objectives: 1) investigating the presence of LFG; 2) assessing LFG human health/safety risks; and 3) remediating LFG constituents. This SOP will provide information to support remedial action decisions regarding LFG controls and compliance with applicable or relevant and appropriate requirements (ARARs).

This standard operating procedure (SOP) outlines the methods used for: 1) installing LFG vapor implants using direct push technology (a.k.a GeoProbe®); and 2) measuring LFG constituents using a portable (direct reading) instrument (e.g., GEM™2000 Plus or similar). The quality of data generated from this SOP using the implant sampling system and direct reading instrument will achieve the objectives outlined above.

This SOP is based on: 1) United Stated Environmental Protection Agency (USEPA) Guidance for Evaluating Landfill Gas Emissions from Closed or Abandoned Facilities (USEPA, 2005); 2) USEPA SOP for Soil Gas Sampling (USEPA, 2001); 3) LANDTEC GEM™2000 Plus Operation Manual (LANDTEC, 2010), and 4) Geoprobe Systems® Direct Push Installation of Devices for Active Soil Gas Sampling and Monitoring (Geoprobe® Systems, 2006). The SOP was adapted from the above references and may be varied as required, depending on site conditions, equipment limitations, procedural limitations, and the objectives defined in the SOP and sampling analysis plan (SAP).

2.0 Soil Vapor Implant Installation Procedure

Ten (10) shallow soil gas probes (a.k.a. vapor implants) will be installed using direct push technology (DPT) to evaluate the presence of LFG constituents. A pilot boring using MacroCores (MC5) Sampling System will be advanced through the bottom of the waste fill layer or to the water table (whichever is shallowest depth) to characterize lithology and to determine/optimize the depth of implant installation to ensure implant is installed above the groundwater table (in the vadose zone). The implants will be installed in a separate hole (approximately five feet away to prevent short circuiting) using 2.25-inch outside diameter (OD) probe rods.

The implant will consist of a stainless-steel wire mesh screen with a threaded fitting on the bottom for anchoring and a fitting at the top to connect to 0.25-inch ID low density polyethylene (LDPE) tubing. The AT86 (GeoProbe®) series screens are 6-inches (152 mm) long, have an outside diameter of less than 0.5-inches (13 mm), and a pore diameter of approximately 0.006-inches (0.15 mm) in the stainless-steel mesh screen. The implants will be installed through the 2.25-inch OD drive rods after they are advanced to the desired depth. An expendable implant anchor/drive point at the lead end of the rod string has a threaded fitting at the upper end and the implant is anchored in place by threading in counterclockwise.

As the rods are retracted with the direct push technology, clean no.1 sand will be poured around the screen to prevent the vapor point screen clogging due to silt, clay, or organic particulates in the surrounding waste/formation. Once the rods are retracted above the screen (6 to 12-inches above screen depending on the depth of hole) a granular bentonite powder will be added to the rod annulus to seal the probe hole. The bentonite may be hydrated when the rod string is completely removed from the ground or conversely, depending on geologic conditions and the depth of probe/implant the bentonite may be chased periodically with small amounts of water to initiate the seal at depth. GeoProbe guidance encourages the larger diameter rods (2.25 or larger) to ensure higher integrity seals and to reduce the likelihood of short circuit with atmospheric air entering the hole versus obtaining formational/landfill gas. The tubing will be capped to prevent venting to the ambient atmosphere until ready for sampling. A conventional 6-inch flush mount well protector, or two-inch polyvinyl chloride (PVC) stickup installed into a small concrete pad (18-inch by 18-inch by 8-inch deep pad).

Once the implants/probes are installed, the sampling implant should be purged at least two volumes using a purge pump or using the pump on the GEM^{TM}2000 Plus during sampling. The purge volume can be determined by the following:

Volume of Cylinder = π x radius^2 x height: Where π = 3.141.

The implants are 0.375-inch (0.95 cm) in diameter and have a length of 6-inches (15.25 cm)

3.141x(0.475 cm)^{2}x15.25 cm = 10.81 cc or 11 ml = volume in implant.

Subtract the implant volume from the total probe hole volume and then multiply the remainder by 0.30 to determine the 30% pore space in the filter media.

2.25-inch (5.715 cm) x 24-inches (61 cm) long results in the following equation:

3.141 x (2.9 cm)^2 x 61 cm = 1611 ml.

(1611 ml -11ml)x 0.30 = 480 ml (pore space in media).

Now add back the total volume of the void in the implant

480 ml+11 ml = 491 ml of void volume in the implant screen and filter media.

To determine the volume in the 0.25-inch (6.4 mm) ID tube use 9.7 ml per foot x tube length (3.7 m).

12 ft x9.7 ml/ft=116.4 ml

Add the probe void volume to the tube volume and multiple by 2 for 2 volumes

=(491ml+116.4 ml) x 2 = 1,214.8 ml

Based on purge rate of 200ml/min = 1,214,8 ml/ 200 ml/min = 6 minutes purge time.

The gas contained in the interstitial spaces of the soil/waste will be pulled through the probe using the pump on the GEM 2000 Plus and the user will monitor the probe using the GA Mode or Gas Analyzer Mode. This mode is used for taking measurements of LFG from Vapor Implants (gas migration probes) and not extraction wells that require the Gas Extraction Mode(GEM). The sample gas flow rate should not exceed 200 ml/min. The pump specifications for the GEM^{TM}2000 Plus are: 1) typical flow is 300 cc/min; 2) flow fail point is 50 cc/min; 3) flow with 200 mbar vacuum is 250 cc/min; and 4) vacuum is 70 inches H_2O.

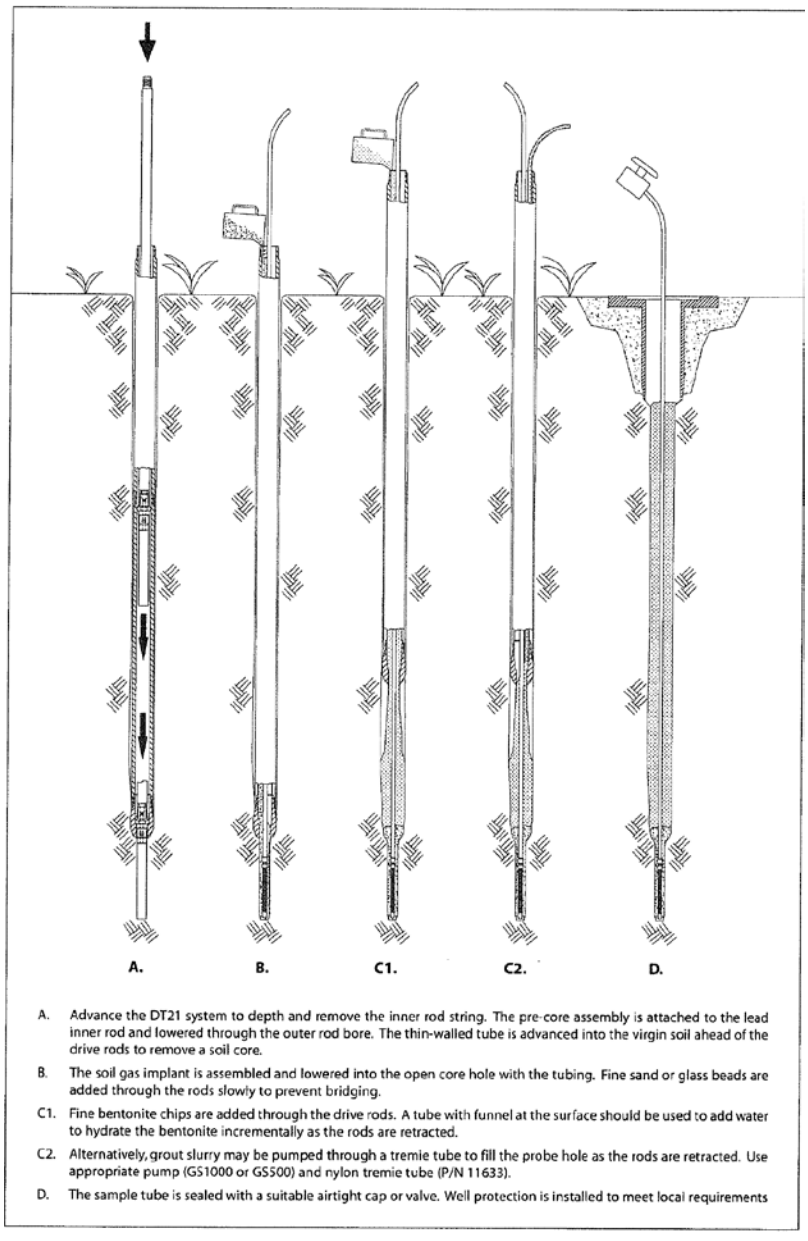


Figure 1. Soil Gas Sampling Implant Installation Schematic (Geoprobe Systems®, 2006).

3.0 GEM^{TM}2000 Plus Gas Analyzer Procedure

At each soil gas implant/probe location, screening-level sampling/analyses will be conducted using a portable (direct reading) instrument GEM^{TM}2000 Plus or similar to determine the presence of methane (CH_4), carbon dioxide (CO_2), oxygen (O_2), carbon monoxide (CO) and hydrogen sulfide (H_2S) and CO.

The following section describes the procedures used for taking LFG measurements using the GEM^{TM}2000 Plus. This SOP requires the User to fully review the Operation Manual prior to using the GEM^{TM}2000 Plus and to ensure that all the items identified in the manual are contained in the shipping package when opened. This includes: the GEM^{TM}2000 Plus instrument, operation manual, registration/warrantee card, 0.25-inch sampling hose with water filter traps, polypropylene male connector, internal particulate filter element, battery charger, DataField CS Software on CD-ROM, and temperature probe (optional but recommended TP-100) among other items.

3.1 Self-Test Procedure

Once the instrument is switched on, the instrument will run a self-test that will take 20 seconds. During the self-test, the instrument's functions will be confirmed (e.g., general operation, pump function, gas flow measurement, and calibration, software version, serial number, etc.) as well as any warnings or errors indicating an operational parameter is out of specification or that the calibration/service date has passed. After the self-test, the User should also confirm that the battery is fully charged or charge the unit accordingly.

3.2 Preliminary User Check Procedure – Calibration and GA Mode

As a preliminary User check, the CH_4, CO_2, and O_2 readings should be auto zeroed, without gas concentration present and the span calibration checked with a known concentration calibration gas. The User shall use the calibrate and zeroing functions (ambient air) for the GEM^{TM}2000 Plus in accordance with the operation manual each day. A normal field calibration usually requires the gas to be running for about two minutes. Spanning channels should be carried out prior to use or when ambient operating temperature changes are greater than plus or minus 20 degrees (CO and H_2S on the GEM^{TM}2000 Plus).

At this time, check to ensure the GEM^{TM}2000 Plus is in the GA Mode by pressing (1) for menu and scroll down to mode of operation and press the return key and highlight Landfill Gas Analyzer by pressing the return key again and it will select the GA mode of operation. Prior to conducting LFG measurement, conduct the following preliminary checks: 1) All necessary ID codes and readings have been uploaded via DataField software; 2) The time and date are correct; 3) The water trap (must always use) has a clean and dry filter fitted; 4) The inlet-port particulate filter is clean and dry; 5) A supply of spare filters is available in case of accidental water blockage or contamination; 6) The battery has a good charge (minimum 25% charge, even if only a few readings are required); 7) The memory has sufficient space available; and 8) The CH4, CO2, and O2 readings have been auto-zeroed, without gas concentration present.

3.3 Normal Operation Screen and Taking Readings

The normal operation screen is the "read gas levels screen," and all operations are carried out from this starting point. During the read gas levels screen the following information is displayed in various boxed sections at this time: 1) Current programmed time and date; 2) Current selected ID code; 3) Pump status; 4) Pump run time; 5) Three main constituent gases – CH_4, CO_2, O_2 (in %); 6) Two minor gases – CO and H_2S (ppm) (GEM™2000 Plus only); 7) Balance gas; 8) Last read time/date (if previous data is in memory); 9) External Gas Pod "Not Fitted" (displays pod type when attached); 10) Peak CH_4 reading (in %) (GA mode only); 11) Lower Explosive Limit (LEL) CH_4 (GA mode only); 12) Current barometric pressure reading; 13) Current relative pressure reading (GA mode only); 14) Gas Pod or Temperature Probe reading (if connected); 15) Battery Charge graph (5 segment, flashes at 20% remaining); and 16) Memory Usage graph (5 segment, flashes at 5% remaining).

When taking sampling reading, the sample readings can be conducted using two modes; 1)Taking Readings with ID; and 2) Take Reading -without ID. Regardless of which the mode, the User needs to purge the instrument between samples using clean air purge.

For each reading, the User will connect the sample tube (with water trap) from the sample point to the inlet port of the instrument, ensuring the connector 'clicks' into place. Then connect the sample tube to the probe sample port. **Do not connect the sample tube to the probe port before connecting to the instrument as this will cause any pressure in the probe to dissipate and a proper pressure reading will not be taken.**

As soon as the connection is made, the relative/static pressure reading will be displayed. No sample is taken from the probe at this time. Once the reading stabilizes and the pump starts, the relative/static pressure reading is stored. The relative/static reading will remain displayed as the pressure last taken.

The pump will run for the pre-programmed time and a countdown timer will be displayed. The pump may be stopped or started at any time by way of the (pump) key. The reading may be stored at any time. When the pump automatically stops this should be used as a prompt to store the reading.

Upon storing the reading, any pre-programmed questions will be displayed for response. This may require a numeric, alphanumeric selectable comment, or exclusive comment answer. A maximum of eight selectable and exclusive comments may be entered.

Disconnect the sample tubing from the probe and proceed to Step 1 for the next probe.

For each reading, the following information will be stored: 1) ID code. (if in ID reading mode); 2 Current time/date; 3) Site data (if entered); 4) All gas readings and balance (CH_4, CO_2, O_2 (, CO and H_2S for the Plus)); 5) LEL CH4; 6) Barometric Pressure; 7) Relative Pressure; 8) Questions/comments 9)Temperature (if temperature probe is connected). The GEM™2000 / GEM™2000 Plus has the facility to automatically display and record the probe temperature via an optional temperature probe (TP-100). When a temperature probe is fitted to the RS232 Communication Socket, the temperature will be displayed in the read gas levels screen and recorded with all other data.

The GEM™2000 Plus will also provide measurements of H2S and CO with following range and resolution.

Summary of GEM™2000 Plus Gas Range and Resolution Information		
Gas Type	**Range (PPM)**	**Resolution**
H_2S	0-50	0.1
CO	0-1,000	1.0

4.0 Interferences and Potential Problems

4.1 Combustible Gas Indicator Measurements

Several factors specific to soil gas can affect the response of a portable gas monitoring instrument (e.g., GEM™2000 Plus). High soil moisture levels, saturated zones, and high concentrations of methane are all soil conditions that could cause instrument interference. "Flow Fail" could occur if the inlet is compromised with a blockage, clogged particulate filter or water trap filter; this can be corrected by removing the blockage and/or changing filters. Unexpected readings may be a result of the unit being out of calibration or clogged filters; this can be corrected by recalibrating instrument and/or changing filters. Methane concentration readings swinging wildly up or down could be the result of RF interference (cell phones); cell phone use should be avoided in the sampling area while taking readings. High oxygen concentration readings could be the result of poorly sealed water trap housing or other connections in the sampling train; all O-rings/seals should be checked. Out of range readings (no value) could result if the parameter concentration is out of the instrument's capabilities (LANDTEC, 2010).

4.2 Factors Affecting the LFG

Concentrations of LFG can be affected by physical and chemical characteristics of the soil and by soil moisture. Soil porosity and permeability will affect the movement of LFG and the recharge rate of the LFG. The movement of LFG through fine textured soil may be very low, thus limiting the sample volume available and the use of the LFG monitoring technique. Movement of LFG can also be limited by a high-water table, perched water table or clay horizon.

4.3 Soil Probe Clogging

A clogged probe is identified by using an in-line vacuum gauge or by listening for the sound of the pump under elevated stress. Probe clogging can be eliminated by water/particulate filter. This must always be used with the GEM™2000 Plus.

4.4 Underground Utilities

Each soil gas sampling location should be surveyed prior to installation of the soil gas probe to ensure no local underground utilities are present to interfere with installation of the probe.

5.0 Equipment/Apparatus

5.1 Direct-Push (Geoprobe) Method

- Tubing: LDPE ¼ ID
- Gas Sampling Cap
- Probe Rods
- Tubing Adaptor
- Threaded Expendable Point Holder
- Expendable Drive Points
- Soil Gas Implants
- No. 1 Sand
- 8-inch Flush Mount
- 2-inch PVC Stickup with Cap
- Concrete and Wood Form For Pad
- O-rings for expendable point holder
- O-rings for adaptor
- O-rings for probe rods
- O-rings for gas sampling cap
- Vacuum Pump
- Tape
- GEM^{TM}2000 Plus
- Water Traps
- Particulate Filters
- Calibration Gas
- Sample documentation materials (logbook, data sheets, etc.)

6.0 Reagents

- Calibration gases
- Deionized water
- Sulfate-free detergent

7.0 Quality Assurance/Quality Control

7.1 Sample Probe Contamination

GEMTM2000 Plus or equivalent) sample probe will be purged after each sample reading by drawing ambient air through the probe using the probe's vacuum pump.

7.2 Combustible Gas Indicator

The combustible gas indicator instrument (GEM™2000 Plus or equivalent) should be calibrated at least once a day using appropriate calibration gases.

8.0 Health and Safety

LFG sampling usually occurs in the minimum Level of personal protective equipment (PPE) for Site entry (usually Level D). Ambient air should be constantly monitored using a PID to obtain background and breathing zone readings during the all soil gas sampling activities. Level of protection should be upgraded as needed.

9.0 References

Geoprobe Systems®, 2006, *Direct Push Installation of Devices for Active Soil Gas Sampling & Monitoring*, May 2006.

LANDTEC, 2010, *GEM™2000/GEM™2000 Plus Gas Analyzer & Extraction Monitor Operation Manual for Serial Numbers less than 10,000*, December 2010.

United States Environmental Protection Agency (UEPA), Guidance for Evaluating Landfill Gas Emissions From Closed or Abandoned Facilities, A-600/R-05/123A, September 2005.

United States Environmental Protection Agency (USEPA), *Standard Operating Procedures, Soil Gas Sampling, SOP: 2042*, April 2001

LFG Screening Log	Date and Time:
Name:	Weather:
Vapor Point No:	
Depth (length):	Diameter:
Screen Interval (length):	Diameter:
Tube (length):	Diameter:
Calculate Purge Volume:	
GEM^{TM}2000 Plus:	Serial No.
Self Test:	Notes:
Calibration:	Notes:
Instrument Purged before Sampling:	Y N Circle.
GEMTM2000 Plus Readings	
CH_4 (percent)	LEL CH_4
CO_2 (percent)	Barometric Pressure
O_2 (percent)	Relative/Static Pressure
CO (ppm)	Temperature
H_2S (ppm)	

Gamma Scanning Survey

RS-022

Revision 1

Reviewed By: _____ Date: Nov 8, 2017

Approved By: _____ Date: Nov 8, 2017

1.0 PURPOSE

The purpose of this procedure is to provide personnel the steps necessary to perform a gamma walkover survey using a GPS system coupled to a sodium iodide detector.

2.0 APPLICABILITY

This procedure provides the requirements and recommended techniques for performing a gamma walkover survey. The procedure will allow Tidewater personnel to characterize large areas for gamma radiation areas of elevated radioactivity.

3.0 PRECAUTIONS, LIMITATIONS AND REQUIREMENTS

3.1.1 Instruments shall be operated in accordance with operating procedures or manufacturer's recommendations, and shall be in current calibration.

3.1.2 This procedure is to be used for the packaging and shipping of limited quantities of radioactive material as defined by IATA. This procedure should not be used for the packaging and shipping of any radioactive material that does not meet the IATA definition of limited quantity.

3.1.3 Only Health Physicists (HPs), Health Physics Technicians (HPTs), or duly authorized personnel who have successfully completed the Basic IATA and Function Specific Training and Testing for Shipments of Limited Quantities of Materials may package and ship limited quantities of radioactive material.

4.0 GPS SET UP AND INITIAL QC

4.1.1 When you receive the GPS unit it should already be configured for the UTM zone you need. You will figure out if your GPS is set up correctly when you submit your first set of data. Corporate GIS personnel will be able to help quickly rectify this problem.

4.1.2 Prior to use establish a daily calibration point to perform daily checks of the GPS system. Make sure that the spot that has been chosen is easily replicable. GPS readings shall be conducted at a designated calibration point each day prior to data collection. This will ensure that the GPS is operating properly and that there is adequate communication with the NAVSTAR satellites.

4.1.3 Turn the GPS on and plug in charged batteries.

4.1.4 At the calibration point click the Terrasync icon on the handset with a pen, or stylus.

4.1.5 Click on the drop down menu in the upper left hand corner of the screen and select Map. It will show a hash mark that represents your location.

4.1.6 Select the zoom button until it shows the scale is at 5 m, or until you can see the hash mark jump around when you are standing still. Click on the hash mark to see your northing and easting.

4.1.7 Take 10 measurements at approximately the same location, and 1 for that days QC check.

5.0 STARTING WALKOVER

5.1.1 Before beginning the walkover, check to ensure that the cables from the GPS unit to the handset, to the antenna, and to the meter are plugged in correctly.

5.1.2 The back packs that hold the GPS unit are large enough for the unit, the meter, and the batteries to fit comfortably. Caution should be exercised when placing the unit into the pack to ensure that the unit is situated such that no damage to the GPS unit or the meter occurs that could result in loss of data.

5.1.3 In general the Trimble TSC2 or TSC3 will be the hand held units that will be used. The following directions apply to either unit.

5.1.4 Turn the unit on.

5.1.5 The screen is a touch screen, use a stylus or a pen, and double click on the icon that is labeled Terrasync.

5.1.6 To create a new data file, select the drop down menu and then select Data.

5.1.7 This provides the option of creating a file name. Name the files in accordance with the naming protocol specified in the applicable work plan.

5.1.8 Choose Line Generic and select OK.

5.1.9 A beeping noise indicates that the unit is collecting data.

5.1.10 The preset logging interval is 5 seconds. To change the logging interval, select logging interval. A drop down will appear. Change the interval from 5 seconds to and the time specified by the work plan and select okay.

5.1.11 To verify that data is recording properly, while recording data:

5.1.12 Select on the drop down menu in the top left corner of the screen, and then select sensor from the drop down menu. In the middle of the screen there will be a string of text that changes at the time interval selected from step 2.3.3.4. The number typically

is preceeded by the letter R and a series of 0. The count per minutes are the numbers that follow the 0's.

5.1.13 To verify coverage while surveying, select Map from the drop down menu. This will bring up the Map screen. Use zoom function to obtain a view that will show the coverage obtained thus far during the survey. If you zoom in to far your map will be distorted, and if you don't zoom in far enough your map will not be a good representation of your coverage. Generally the scale in the lower left hand corner should be around 100 or 50.

*Note: *It is important to periodically switch between the sensor screen and the map screen. During the walkover something can happen to the meter while it is in the back pack and you will not know about it if you do not check out the sensor screen. It is also important to check your map to ensure you are getting quality coverage.*

5.1.14 While performing the survey check the life of the batteries. With newer Trimble units the TSC 2 and 3's, they "should" save the data when they run out of battery life.

6.0 PERFORMING WALKOVER

6.0.1 Before beginning the walkover, review the areas to be surveyed for the following:

- Terrain,
- Size,
- Elevation changes, etc.

6.0.2 To ensure adequate coverage when surveying large areas markers should be used to delineate survey lanes. Cones, pin flags, tape measures, or delineators, can be used as a visual guide to help establish straight lines. Place markers at each end of the survey lane If you are standing at one cone and cannot see the other cone you are trying to cover an area that is to large. The smaller distance you walk between two points the tighter and better your coverage will look.

6.0.3 The width between paths should be about one meter. When the survey lane is completed, move the marker one meter to the side to set it for the next survey lane. The typical speed for a gamma walkover survey is a half a meter per second. The actual speed will be specified in the work plan based upon the detection level and anticipated background of the area to be scanned. Survey pace can be maintained by using the beeps of the Trimble handset. The unit will beep every second. A half meter is typically obtained by taking about half of a normal stride when walking.

6.0.4 While walking swing your arm slowly in a “pendulum” type motion. Try to cover as much of your one meter wide path as possible. Keep the detector at the height specified in the work plan to maintain the correct MDC. (Typically a height of approximately 4” of the ground in order to maximize the scan MDC).

6.0.5 Utilize the volume on the meter to to distinguish when the count rate increases from normal levels. Follow the guidance from the work plan for identifying or pausing when increased count rates are identified.

6.0.6 In order to close a file under the map screen, or in the data screen:

6.0.7 In the data screen there will be a button that has a red square on it that means stop. Click that button, and it will stop recording data. A dialog box will appear and asks “Do you want to continue” click Ok.

6.0.8 The handset will still continue to beep like it is still collecting data. , click the X in the top of the right hand corner of the next screen and close out of Terrasync.

6.0.9 Uploading Files and Collecting Data

6.0.10 before beginning the survey determine the size of the file. Record data for a specified amount of time, so files will be a manageable size to upload and possibly email.

6.0.11 To upload your files off of your handset to a computer:

6.0.12 Connect GPS handset to the computer

6.0.13 On your computer open Pathfinder

6.0.14 Click “Utilities”, “Data Transfer”

6.0.15 You should see an icon of a serial pot with a green circle around a check mark (as opposed to the red x) indicating that the GPS is talking to your computer and vice versa. If you don’t see this, try re-connecting all of the cords

6.0.16 Click on the “receive” tab. This allows you to receive files from the GPS receiver.

6.0.17 Click “Add”, then “Data file”.

6.0.18 Click on the data file name you created and press “Open”.

6.0.19 Click “Transfer all”

In-Situ Gamma Spectroscopy

Work Instruction WI-002

TIDEWATER INC
ENGINEERS / SCIENTISTS / PROGRAM MANAGERS

1.0 Purpose

This work instruction (WI) provides an operating procedure for the Ortec TransSpec.

2.0 Discussion

The TransSpec provides the capability to perform gamma spectral analysis in the field. The unit operates off battery and displays results of the spectral analysis immediately to the operator in the field. The TransSpec can be used in conjunction with the Genie 2000 and Geometry Composer software to provide a detailed gamma peak analysis and quantification.

This WI is applicable to all forms of field in situ gamma spectroscopy, including non-destructive assay of waste containers or components and characterization of soils and other environmental media. There are seven main steps involved in operating the TransSpec system. Each of the steps are covered in the Procedure section of this instruction (Section 5.0):

- Cooling the detector
- Set-up of the detector, accessories/equipment, and electronics
- Acquiring a spectrum
- Calibration of the system
- Analysis of the data
- Reporting data, and
- Quality Assurance (QA)/Quality Control (QC)

3.0 Procedure

3.1. Plug in and supply power to the power box as shown in figure 1. This will keep the Transpec cool and keep the battery charged. If there is no power available skip this step. The Transpec will still run internal cooling and you will have a few hours of battery life.

FIGURE 1, POWER SUPPLY CONNECTION

FIGURE 2, POWER CONNECT TO TRANSSPEC

3.2. On the opposite end of the power box there is a fixed cable coming from the box. Connect the free end with the connector to the Transpec. This supplies power to the Transpec. If no power available skip this step. See Figure 2.

3.3. Remove the 2 wing nuts on the front of the TransSpec to access the memory card. Ensure there is a card available and insert it into the TransSpec.

3.4. Press the power button on the top of the TransSpec to turn the unit on.

3.5. This is the first screen that appears when the Transpec is turned on. Don't touch anything. Give the instrument some time to boot up and it should automatically load the Ortec software. See figure 3.

FIGURE 3, START UP SCREEN

3.6. The screen shown in figure 4 will appear next. From here click on the "Menu" button in the bottom right. (there is a stylus located on the front of the instrument. Under where the SD card is placed. It is magnetically stored in this location.)

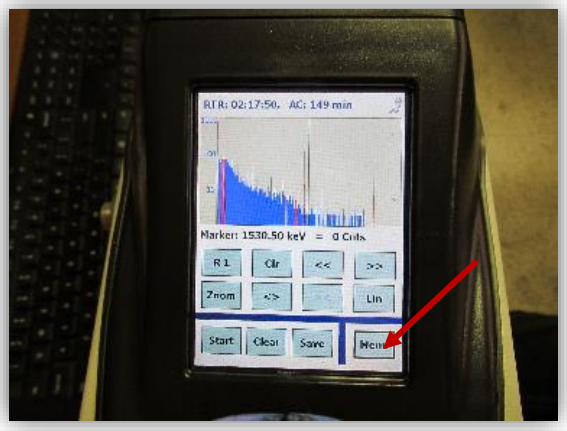


FIGURE 4, PROGRAM SCREEN

3.7. First click on "Sample Desc" shown in figure 5 to be able to name/describe what it is that is being analyzed. Type the name/description of the subject being analyzed using the on screen keyboard. When finished click "set" then click the "back" button (Figure 6)

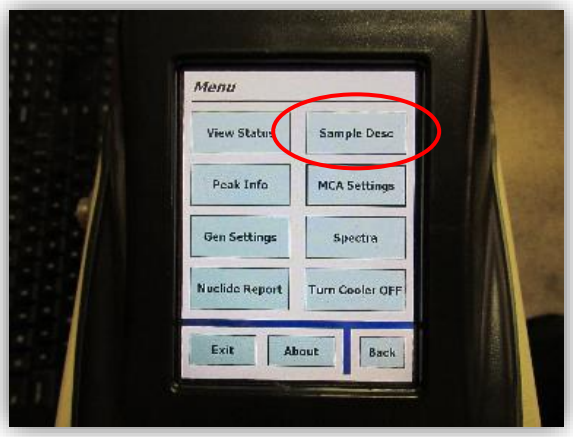


FIGURE 5, SAMPLE DESC SCREEN

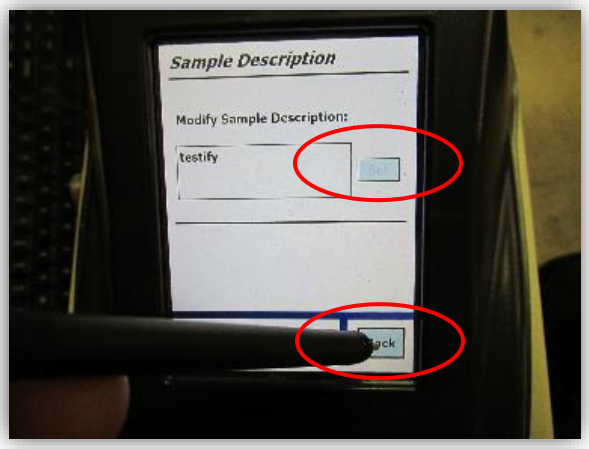


FIGURE 6, SAMPLE DESCRIPTION

3.8. The program will return to the menu screen. Click the "MCA Settings" button. Then click on the "Presets" button. Then select, “Live Time”.

3.9. Enter the time (in seconds) that you want to analyze the subject using the on screen keyboard. When finished click the "Set" button. See Figure 7. This will return you to the Menu screen.

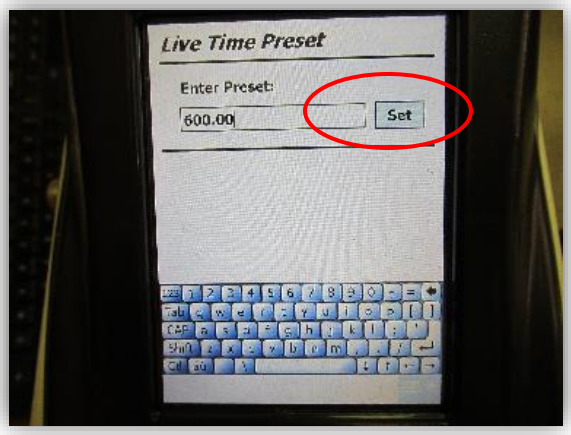


FIGURE 7, LIVE TIME PRESET

3.10. Select the “Spectra” button.

3.11. Ensure that the sample description looks as you entered it and that the data location is where you want to store the data. (i.e. if you are using a SD card). Click the "back" button until you are back at the original screen. See Figure 8.

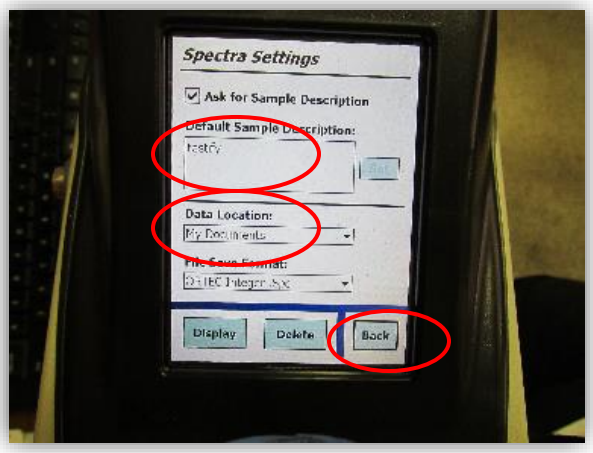


FIGURE 8, SPECTRA SCREEN

3.12. From this spectra screen click the "start" button to start the count. The "RTR" at the top left of the screen will count down. When the instrument is finished counting click "save". To perform another count with the same settings click "clear" and then "start". Figure 9.

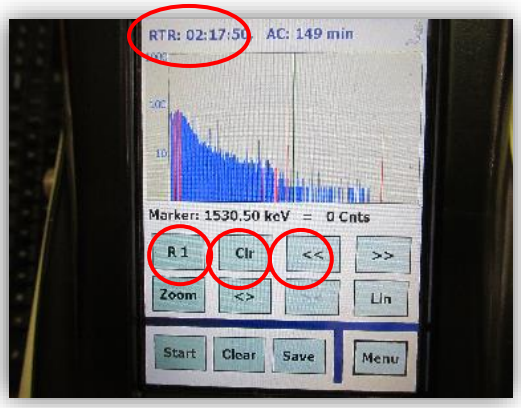


FIGURE 9, SPECTRA SCREEN

3.13. Perform a Quality Control count using a cesium 137 (Cs-137) button source placed 25 centimeters (cm) or 10 inches (in) from the face of the detector.

3.14. If the Cs-137 peak is not in the correct channel, notify the Project HP to perform an energy calibration.

3.15. Press the Enter button and select Save from the dialog box that appears to save the QC spectrum.

3.16. Place the item to be analyzed 10 inches from the face of the detector. Note that the Project HP may adjust the distance based upon the survey results.

3.17. Using the Spec Worksheet, enter the information regarding the item (source) and any shielding between the source and the detector face.

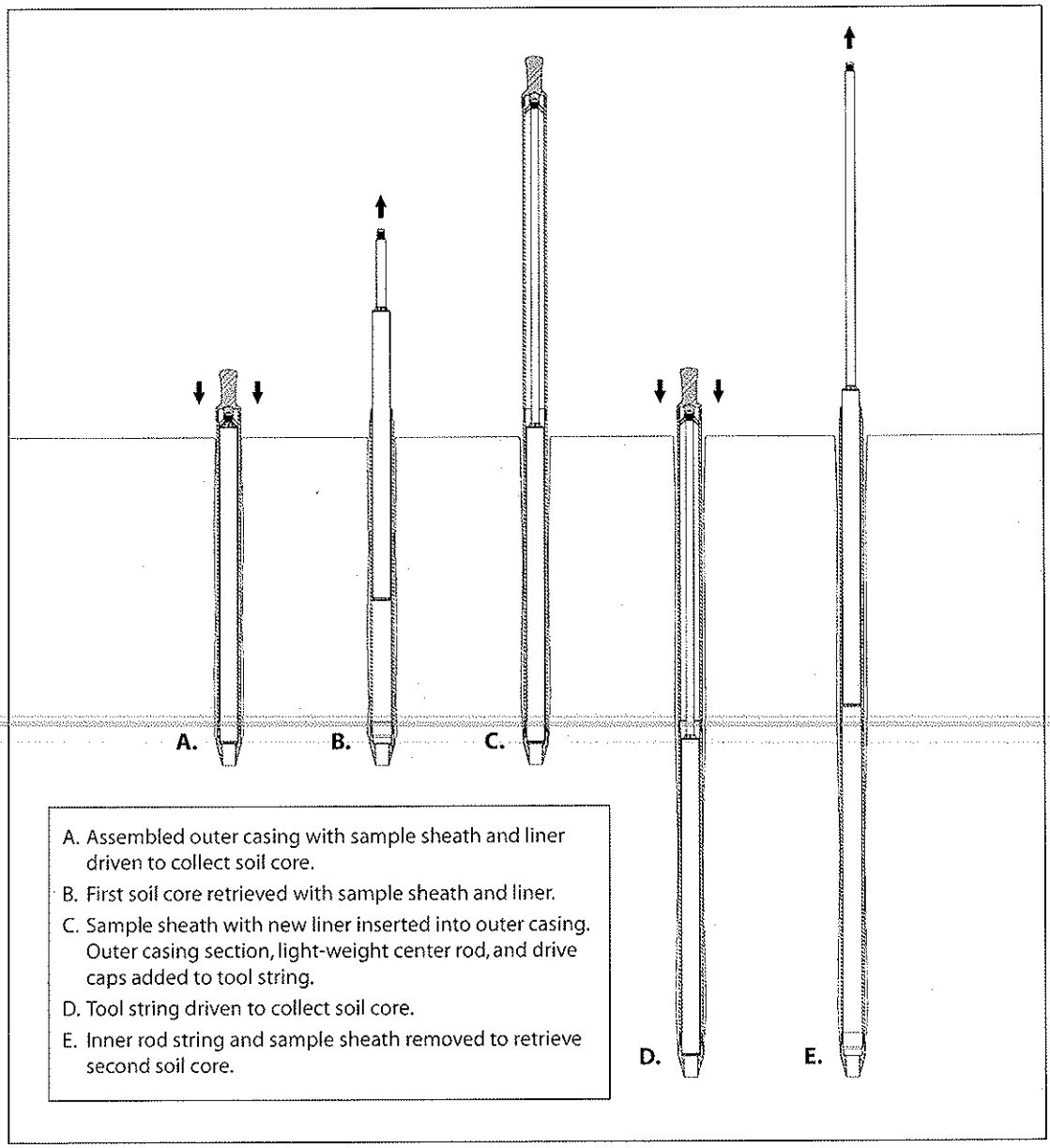
3.18. Obtain radiation dose rate and/or count rate information from the source. Record the data on the Spec Worksheet.

GEOPROBE® DT325 DUAL TUBE SAMPLING SYSTEM

STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3138

PREPARED: November, 2006



Collecting soil cores with the DT325 Dual Tube Sampling System.

1.0 Objective

The objective of this procedure is to collect a representative soil sample at depth through an enclosed casing and recover it for visual inspection and/or chemical analysis.

2.0 Background

2.1 Definitions

Geoprobe®*: A brand name of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, soil conductivity and contaminant logging, grouting, and materials injection.

**Geoprobe® and Geoprobe Systems® are registered trademarks of Kejr, Inc., Salina, Kansas.*

DT325 Dual Tube Sampling System: A direct push system for collecting continuous core samples of unconsolidated materials from within a sealed casing of Geoprobe® 3.25-inch (83 mm) OD probe rods. Samples are collected and retrieved within a sample sheath and liner that is threaded onto the leading end of a string of Geoprobe® 1.25-inch (32 mm) OD light-weight center rods and inserted to the bottom of the outer casing. Collected samples measure up to approximately 2,600 ml in volume in the form of a 1.85-inch x 59-inch (47 mm x 1499 mm) core when using common equipment options.

Liner: A 2.1-inch (53 mm) OD thin-walled, PVC tube that is inserted into the outer casing on the leading end of the inner rod string for the purpose of containing and retrieving core samples. Liners are available in two configurations; a simple open tube or a tube with a core catcher permanently attached to the leading end. Nominal liner lengths include 1 meter, 48 inches, and 60 inches.

***Nominal liner length identifies the length of tools with which the liner is used. The actual end-to-end lengths of the various DT325 liners will differ from the specified nominal lengths.*

Core Catcher: A dome-shaped device positioned at the leading end of a liner to prevent loss of collected soil during retrieval of the liner and soil core. Flexible fingers at the top of the core catcher are pushed outward by soil entering the liner during advancement of the tool string. As the filled liner is subsequently retrieved, the fingers of the core catcher move back inward, effectively closing off the end of the liner and limiting soil loss. The core catcher designed for the DT325 system is made of PETG material and is permanently fused to the liner.

2.2 Discussion

Dual tube sampling gets its name from the fact that two sets of probe rods are used to retrieve continuous soil core samples from the subsurface. One set of rods is driven into the ground as an outer casing (Fig. 2.1). These rods receive the driving force from the hammer and provide a sealed casing through which soil samples may be recovered. The second, smaller set of rods are placed inside the outer casing with a sample liner attached to the leading end of the rod string (Fig. 2.1). These smaller rods hold the liner in place as the outer casing is driven to fill the liner with soil. The inner rods are then retracted to retrieve the full liner.

Standard Geoprobe® 3.25-inch OD probe rods provide the outer casing for the DT325 Dual Tube Soil Sampling System. A cutting shoe is threaded into the leading end of the rod string. When driven into the subsurface, the cutting shoe shears a 1.75- or 1.85-inch OD soil core (depending on cutting shoe option) which is collected inside the casing in a clear plastic liner.

The second set of rods in the DT325 dual tube system are Geoprobe® 1.25-inch OD light-weight center rods. A sample sheath with PVC liner is attached to the end of these smaller rods and then inserted into the casing. The 1.25-inch light-weight center rods hold the sample sheath tight against the cutting shoe as the outer casing is driven to collect the soil core. Once filled with soil, the sample sheath and liner are removed from the bottom of the outer casing by lifting out the 1.25-inch center rod string.

The outer, 3.25-inch probe rods provide a cased hole through which to sample. The main advantage of sampling through a cased hole is that there is no side slough to contend with. In addition, the outer casing effectively seals the probe hole when sampling through perched water tables. These factors mean that sample cross-contamination is eliminated. The DT325 sampling system is therefore ideal for continuous coring in both saturated and unsaturated zones.

Solid Drive Tip

A Solid Drive Tip (28509 or 27763) can be placed on the leading end of the inner 1.25-inch rod string in place of a sample sheath and liner (Fig. 2.2). When installed in the outer casing, the drive tip firmly seats within the cutting shoe and effectively seals the tool string as it is driven into the subsurface. This enables the operator to advance the outer casing to the bottom of a pre-cored hole or through undisturbed soil to reach the top of the sampling interval.

Grouting

The DT325 system allows bottom-up grouting through the primary tool string. This means that a cement or bentonite grout mix can be pumped through the outer casing as it is withdrawn from the ground. This is in contrast to most other soil samplers which require driving a second set of tools back down the probe hole in order to deliver the grout mix.

Monitoring Well Installation

An expendable cutting shoe enables the operator to install a Geoprobe® prepacked screen monitoring well through the outer casing of the DT325 Dual Tube System. After the collection of continuous soil cores to the desired depth, prepacked screens can be inserted to the bottom of the outer casing on the leading end of a PVC riser string. The well is finished, complete with grout barrier, bentonite well seal, and a high-solids bentonite slurry/neat cement grout, during retrieval of the outer casing.

Groundwater and Soil Gas Profiling

The DT325 system can be combined with a simple screen to conduct vertical profiling of groundwater or soil gas quality through the leading end of the outer casing. Utilizing the Geoprobe® Groundwater Profiler (Geoprobe® 2003) with the DT325 system provides access to groundwater or soil gas (Geoprobe® 2006) at multiple depths in a single push. In addition, hydraulic conductivity testing may be performed through the groundwater profiler tool string with the Geoprobe® Pneumatic Slug Test Kit (Geoprobe® 2005).

A DT325 Drivable Profiler Head (21379) adapts the groundwater profiler to the inner 1.25-inch rod string of the DT325 system. The profiler head is machined to fit within the DT325 cutting shoe. An O-ring on the profiler head provides a watertight seal to prevent groundwater flow into the outer casing when conducting slug tests through the groundwater profiler screen.

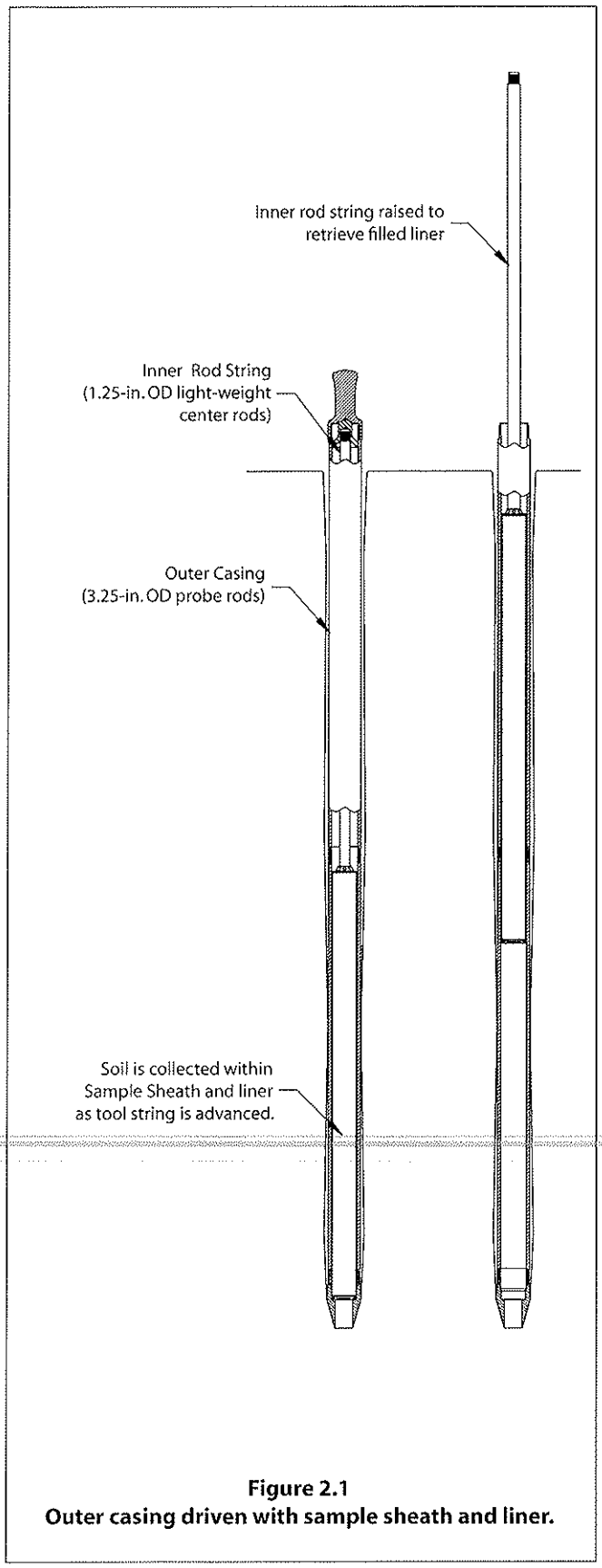


Figure 2.1
Outer casing driven with sample sheath and liner.

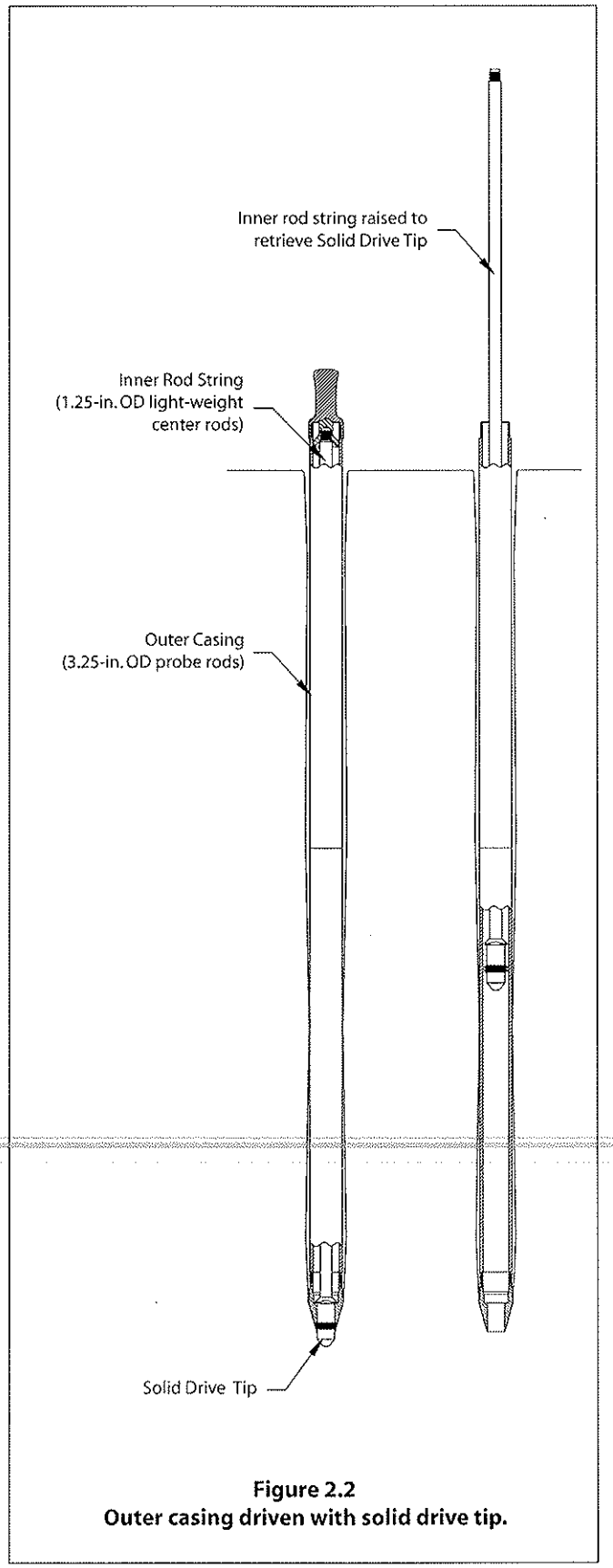


Figure 2.2
Outer casing driven with solid drive tip.

3.0 Tools and Equipment

The following equipment is required to operate the DT325 Dual Tube Sampling System. Refer to Figure 3.1 for identification of the specified parts.

DT325 Sampler Parts*	Quantity	Part Number
DT325 Sheath Drive Head	-1-	10212
DT325 Sample Sheath, 72-in. length	-1-	26805
DT325 Sample Sheath, 60-in. length	-1-	26719
DT325 Sample Sheath, 48-in. length	-1-	27711
DT325 Sample Sheath, 1-m length	-1-	27712
DT325 Cutting Shoe, standard, 1.85-in. ID	-1-	28508
DT325 Cutting Shoe, optional, 1.75-in. ID	-1-	26720
DT325 Expendable Cutting Shoe Holder	-1-	28339
DT325 Expendable Cutting Shoe, 1.75-in. ID	-1-	28341
DT325 Solid Drive Tip, for standard (28508) cutting shoe	-1-	28509
DT325 Solid Drive Tip, for optional cutting shoe (28341) and expendable cutting shoe (26720)	-1-	27763
Replacement O-rings, for DT325 solid drive tips (28509 and 27763), pkg. of 25	Variable	13942
DT325 Liner Retainer	-1-	26718
O-rings, for DT325 liner retainer (26718), pkg. of 25	Variable	28379
DT325 Liner Retainer, without O-ring groove	-1-	31039
DT325 Liner Retainer Wrench	-1-	27838

DT325 Liners and Accessories	Quantity	Part Number
DT325 Liner Spacer, pkg. of 21	Variable	29609K
DT325 Liner Spacer Head	-1-	29358
DT325 PVC Liner, 60-in. length, box of 43	Variable	DT3260K
DT325 PVC Liner, 48-in. length, box of 43	Variable	DT3248K
DT325 PVC Liner, 1-m length, box of 43	Variable	DT3239K
DT325 PVC Liner with Core Catcher, 60-in. length, box of 43	Variable	27813
DT325 PVC Liner with Core Catcher, 48-in. length, box of 43	Variable	27814
DT325 PVC Liner with Core Catcher, 1-m length, box of 43	Variable	27815
DT325 Vinyl End Caps, pkg. of 84 (42 pair)	Variable	17762
DT325 Liner Cutter	-1-	26155
Universal Liner Holder	-1-	22734

Probe Rods and Accessories*	Quantity	Part Number
GH60 Threadless Drive Cap, 3.25-in. rods**	-1-	9742
Pull Cap, 3.25-in. rods	-1-	13257
Probe Rod, 3.25-in. OD x 60-in. length	Variable	9040
Probe Rod, 3.25-in. OD x 48-in. length	Variable	10594
Probe Rod, 3.25-in. OD x 1-m length	Variable	13925
Probe Rod, 3.25-in. OD x 12-in. length	-1-	13672
Replacement O-rings, for 3.25-in. probe rods, pkg. of 25	Variable	9960
Centering Drive Cap, 1.25-in. rods	-1-	12943
Pull Cap, 1.25-in. rods	-1-	AT1204
Light-Weight Center Rod, 1.25-in. OD x 60-in. Length***	Variable	27600
Light-Weight Center Rod, 1.25-in. OD x 48-in. Length***	Variable	21900
Winch Pull Plate, 1.5-in. rods (also works with 1.25-in. OD rods)	-1-	18274
Adjustable Rod Clamp	-1-	27216

Optional Accessories	Quantity	Part Number
DT325 Adapter for Hydraulic Liner Extruder	-1-	24959
DT325 Plunger for Hydraulic Liner Extruder	-1-	23977
Rod Grip Handle, GH60 Hammer, 3.25-in. rods	-1-	9757
Rod Grip Handle, GH60 Hammer, 1.5-in. and 1.25-in. rods	-1-	15554
Rod Wiper Donuts, 3.25-in. Rods	-1-	27194
Rod Wiper Weldment	-1-	23633

* Select DT325 Sample Sheath and liner lengths to match length of probe rods.
** A 3.25-inch probe rod drive cap is also available for use with GH40 Series hammers.
*** 1.25-inch OD probe rods may be substituted for Light-Weight Center Rods.

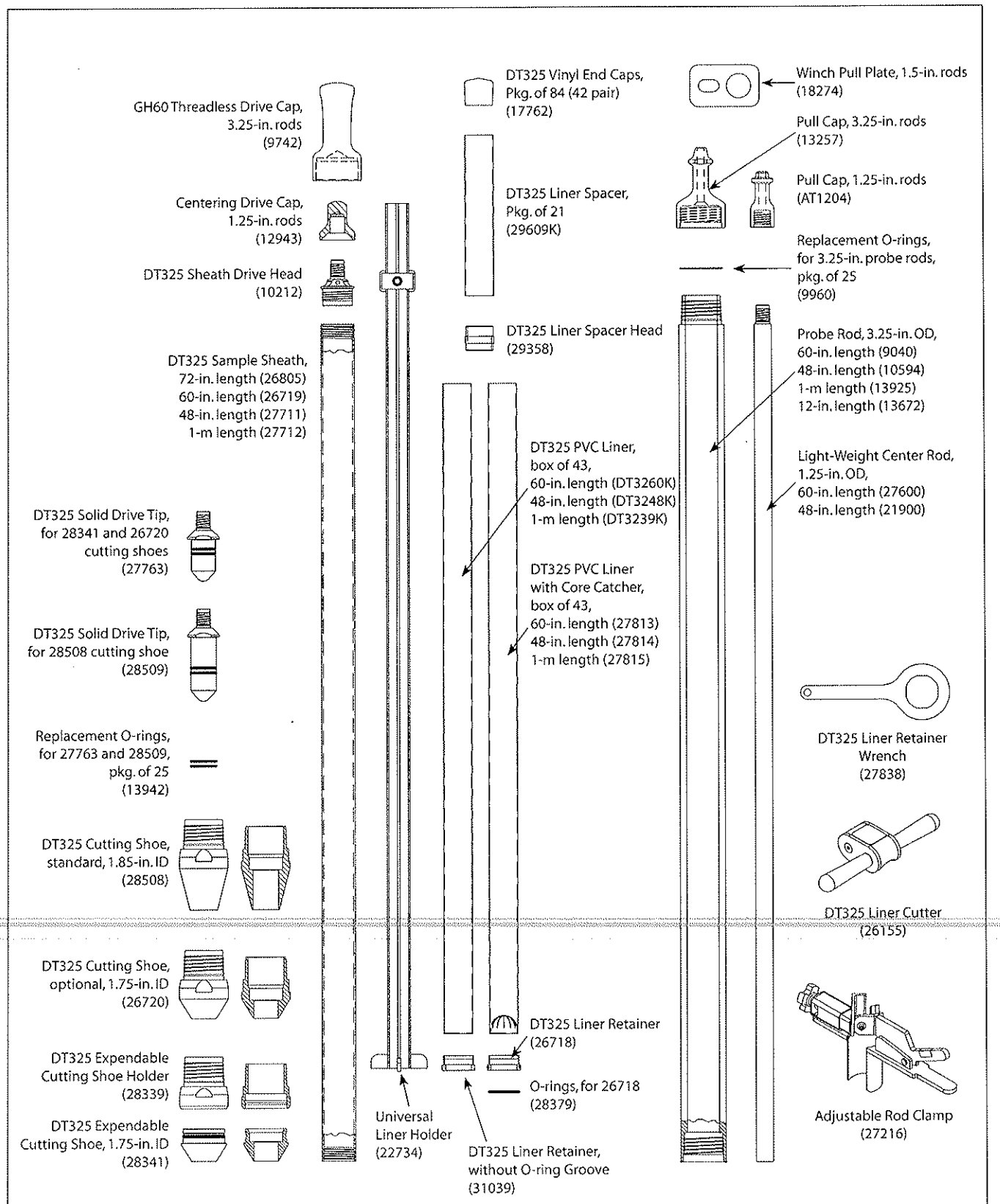


Figure 3.1
DT325 parts and accessories.

3.1 Tool Options

This section identifies the specific tool options available for use with the DT325 Dual Tube System. Refer to Figure 3.1 for illustrations of the specified parts.

Probe Rods

Standard Geoprobe® 3.25-inch (83-mm) OD probe rods are utilized for the outer casing of the DT325 Sampling System. Nominal rod lengths include 1 meter, 48 inches, and 60 inches. The specific length of rods may be selected by the operator and will determine the length of tooling for the rest of the DT325 system.

1.25-inch Light-Weight Center Rods

1.25-inch Light-weight center rods (1.25-inch / 32-mm OD) are recommended for the inner rod string of the DT325 system when utilizing an outer casing of 48- or 60-inch long rods. Choose the light-weight rod length that matches the length of rods used for the outer casing (48-inch light-weight rods with 48-inch outer casing, etc.). Currently, 1.25-inch light-weight center rods are not available in 1-meter lengths. Standard Geoprobe® 1.25-inch x 1-meter probe rods must be used with 3.25-inch x 1-meter outer casing.

A weight reduction of up to 64% is provided by the 1.25-inch light-weight center rods over standard 1.25-inch probe rods. As a result, considerably less energy is expended when retrieving the light-weight center rods from within the outer casing during operation of the DT325 Dual Tube System.

Sample Sheaths

A steel sample sheath supports the weight of the inner rods to protect the sample liner from damage while advancing the DT325 tool string. The liner is placed within the sheath and secured with a drive head at the top of the sheath and a liner retainer at the bottom. The assembled sheath with liner is inserted to the bottom of the outer casing on the leading end of the inner rod string (light-weight rods). After advancing the entire tool string one sample interval, the inner rods and sample sheath are retrieved to recover the soil core.

Sample sheaths are available in nominal lengths of 1 meter, 48 inches, 60 inches, and 72 inches. Sample sheath length is generally matched to the length of the probe rods selected for the outer casing. However, a DT325 Liner Spacer (29609K) and DT325 Liner Spacer Head (29358) allow use of 48-inch liners with a 60-inch Sample Sheath (26719) and 60-inch liners with a 72-inch Sample Sheath (26805).

Sample Liners

Sample liners are made of a heavy-duty clear PVC for convenient inspection of the soil sample. Liners are available either as a simple, open tube or with an intergral core catcher. Utilize the core catcher liners when sampling flowing sands, noncohesive soils, extremely dry soils, or any other materials that fall from the liner during retrieval.

Nominal liner lengths include 1 meter, 48 inches, and 60 inches with an OD of 2.1 inches (53 mm). Under "normal" sampling conditions, liner length should correspond to the length of probe rods used for the outer casing. Certain sampling conditions can cause over-filled liners which may lead to problems removing the liner and soil core from the sample sheath. For these special conditions, utilize a Liner Spacer (29609K) and DT325 Liner Spacer Head (29358) to provide additional room above the liner for the excess soil (Fig. 3.2). The liner spacer and liner spacer head must be used with either a 48-inch liner in a 60-inch Sample Sheath (26719) or a 60-inch liner in a 72-inch Sampler Sheath (26805). With the tool string only advanced the length of the liner, the liner spacer remains free to accept excess soil that may otherwise overfill the liner.

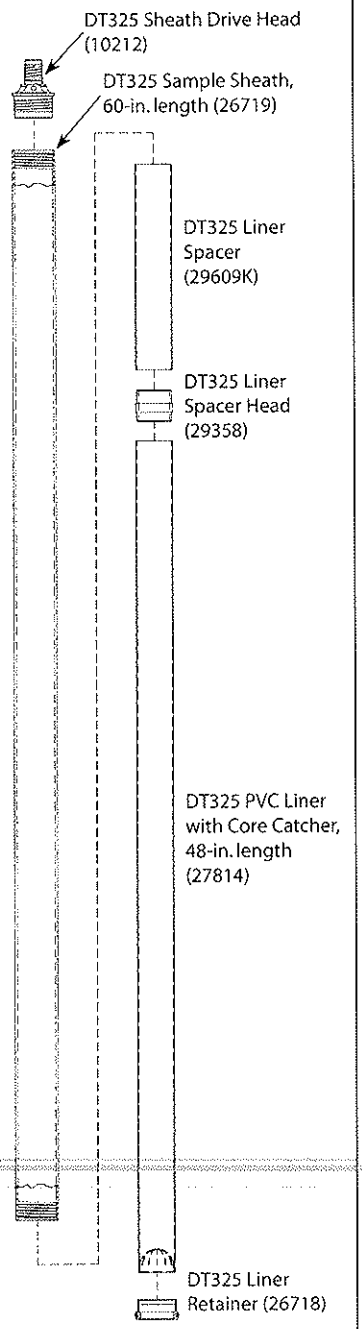


Figure 3.2
Common tooling configuration for use of DT325 Liner Spacer.

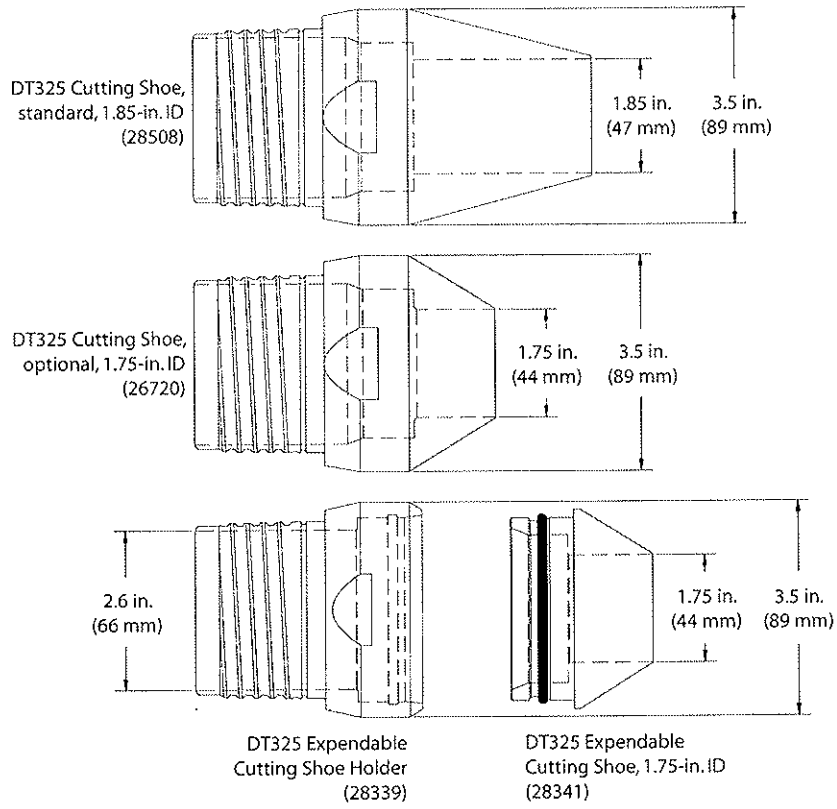


Figure 3.3
Cutting shoe options for the DT325 Dual Tube Sampling System.

Cutting Shoes

Three cutting shoes are available for use with the DT325 Dual Tube System (Fig. 3.3). The DT325 Standard Cutting Shoe (28508) and DT325 Optional Cutting Shoe (26720) thread into the leading end of the 3.25-inch probe rods and are recovered after sampling. Dimensions for the standard cutting shoe are 1.85 inches (47 mm) ID and 3.5 inches (89 mm) OD. The optional cutting shoe also has an OD of 3.5 inches (89 mm), but the ID is only 1.75 inches (44 mm). The standard cutting shoe is ideal for sampling plastic clays and saturated sands while the optional cutting shoe is designed for use in formations where a smaller-diameter soil core is beneficial to sample recovery.

The DT325 sampling system may also employ an expendable cutting shoe (Fig. 3.3). In this arrangement, a DT325 Expendable Cutting Shoe Holder (28339) is threaded into the leading end of the outer casing. A DT325 Expendable Cutting Shoe (28341) is then inserted into the holder. Upon completion of soil sampling, the outer casing is withdrawn slightly. The expendable cutting shoe is knocked from the holder, leaving an open casing through which a prepacked screen monitoring well may be installed. Dimensions for the expendable cutting shoe are the same as the optional cutting shoe (ID = 1.75 in. (44 mm) and OD = 3.5 in. (89 mm)).

4.0 Operation

4.1 Decontamination

Before and after each use, thoroughly clean all parts of the soil sampling system according to project requirements. Parts should also be inspected for wear or damage at this time. During sampling, a clean new liner is used for each soil core.

4.2 Operational Overview

The DT325 Soil Sampling System is designed to collect continuous soil cores. Sampling may begin either from ground surface or a predetermined depth below ground. Once sampling begins, consecutive soil cores are removed as the outer casing is advanced to greater depths

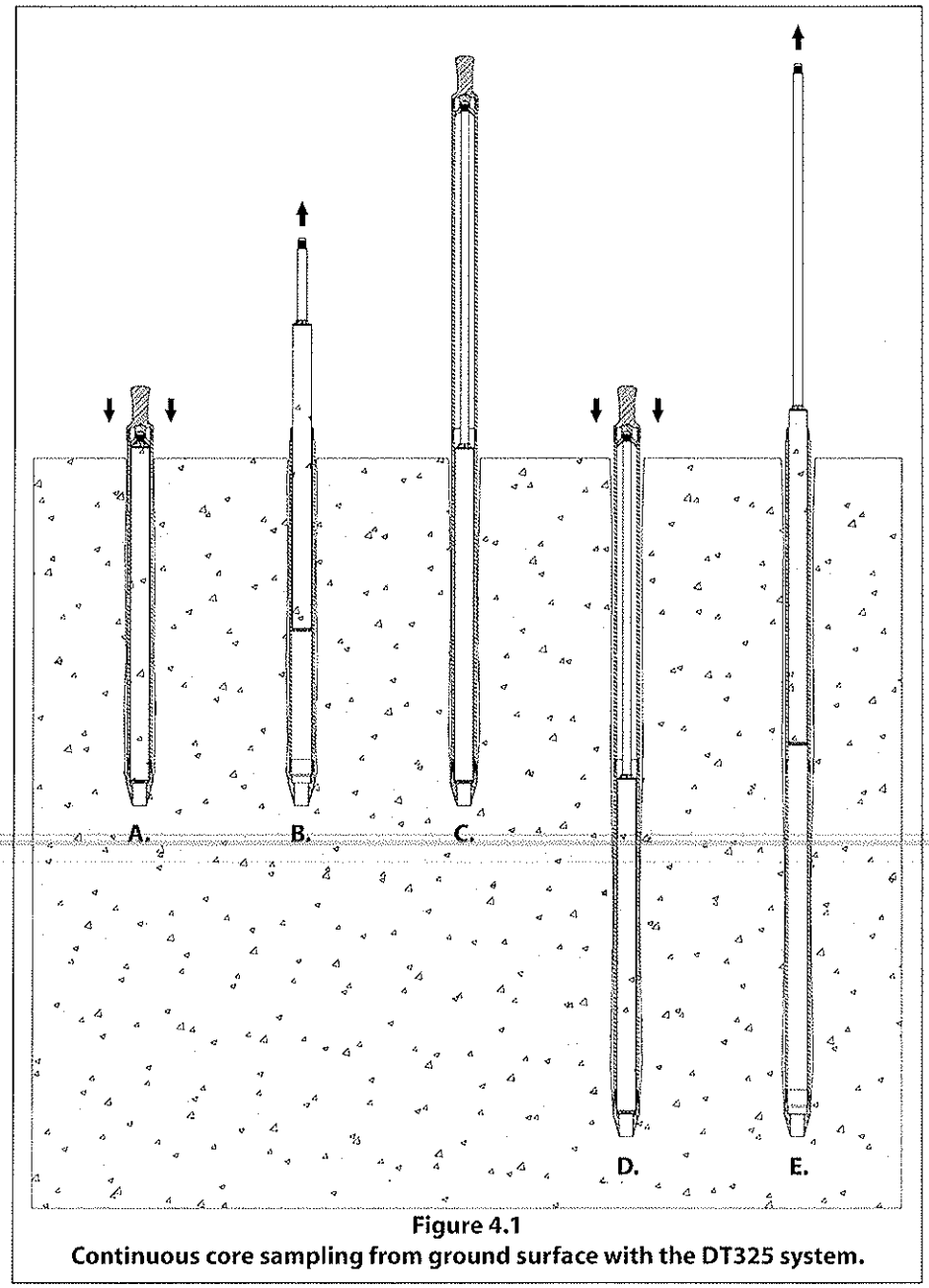


Figure 4.1
Continuous core sampling from ground surface with the DT325 system.

When sampling is to begin at the ground surface, the first soil core is generally collected using a liner with core catcher to maximize sample recovery (Fig. 4.1-A). This is especially true when the first core is composed of dry, loose soil. Upon retrieval of the first liner and soil core (Fig. 4.1-B), a new liner is loaded into the sample sheath and inserted to the bottom of the outer casing on the end of an inner rod. A section of outer casing is added to the tool string (Fig. 4.1-C) and the entire tool string is driven to fill the liner with soil (Fig. 4.1-D). The sample sheath and filled liner are removed from the outer casing to retrieve the second soil core (Fig. 4.1-E). A new liner is placed in the sample sheath and the process is repeated for the entire sampling interval.

When the sampling interval begins at some depth below ground surface, a DT325 Solid Drive Tip is installed in the outer casing and the entire assembly is driven from ground surface directly through undisturbed soil (Fig. 4.2-A). This enables the operator to reach the top of the sampling interval without stopping to remove unwanted soil cores. Once the interval is reached, the solid drive tip is removed (Fig 4.2-B) and sampling continues as described in the preceding paragraphs (Fig. 4.2-C, Fig. 4.2-D, and Fig. 4.2-E).

Specific instructions for assembly and operation of the DT325 Sampling System are given in the following sections.

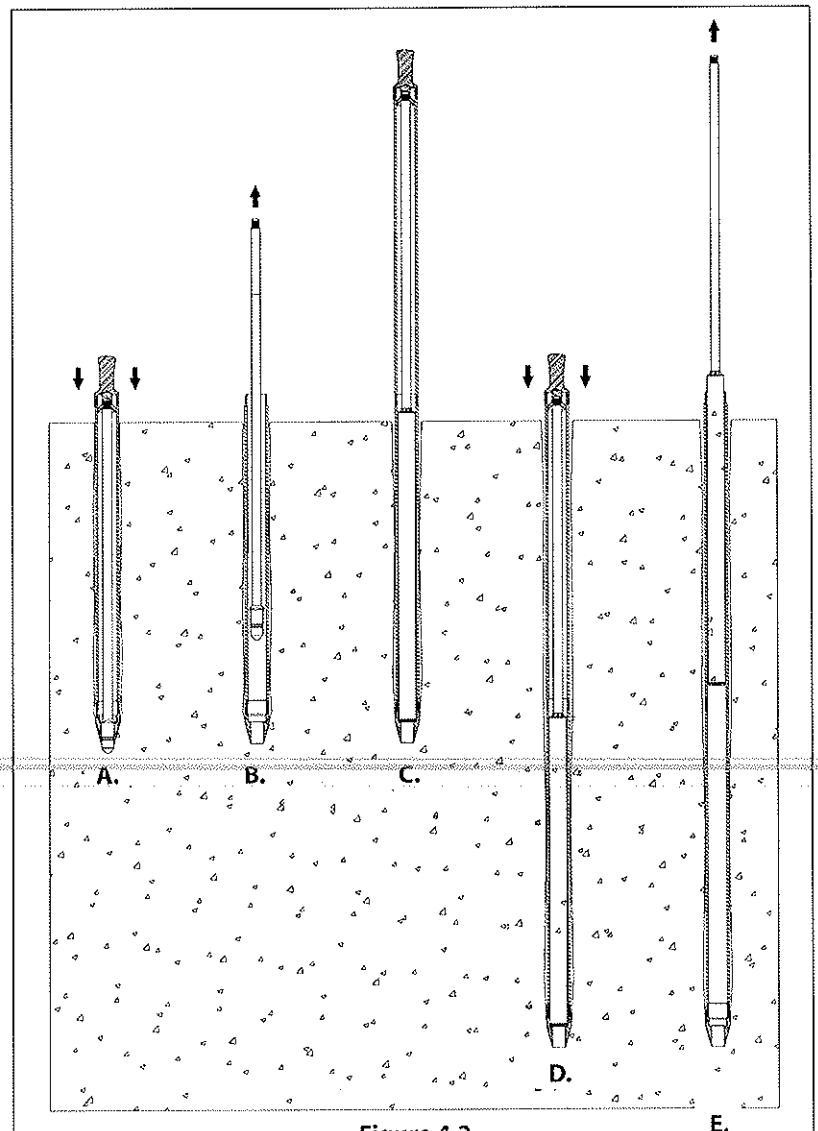


Figure 4.2
Using a solid drive tip to advance the DT325 outer casing through undisturbed soil before collecing soil cores.

4.3 Assembling and Driving the Outer Casing Using a DT325 Solid Drive Tip

A solid drive tip enables the operator to advance the outer casing to the bottom of a pre-cored hole or through undisturbed soil to reach the top of the sampling interval. The outer casing is assembled first, followed by the 1.25-in. light-weight center rod system with a solid drive tip. Step by step instructions are listed below.

1. When using a DT325 Standard (28508) or Optional (26720) Cutting Shoe, install an O-ring (9960) at the base of the theads as shown in Figure 4.3. If using an expendable cutting shoe, install an O-ring (9960) on the DT325 Expendable Cutting Shoe Holder (28339) and one o-ring (6834) on the DT325 Expendable Cutting Shoe (28341).

2. Thread the DT325 Cutting Shoe or DT325 Expendable Point Holder onto the leading end of a 3.25-inch OD Probe Rod. Completely tighten the cutting shoe or cutting shoe holder using a pipe wrench.

3. Install an O-ring (13942) in both grooves of the DT325 Solid Drive Point (27763 or 28509).

4. Thread the solid drive point into the female end of a 1.25-inch light-weight center rod.

5. Lubricate the O-rings on the solid drive point with a small amount of deionized water. Insert the point and probe rod into the outer casing until the point partially extends from the bottom of the cutting shoe.

6. Place a Centering Drive Cap (12943) on top of the 1.25-inch light-weight center rod and a GH60 Threadless Drive Cap (9742) onto the 3.25-inch probe rod (outer casing) as shown in Figure 4.5.

7. Raise the probe unit hammer assembly to its highest position by fully extending the probe cylinder.

8. Position the assembled outer casing section directly under the hammer with the cutting shoe centered between the toes of the probe foot. The assembled outer casing section should now be parallel to the probe derrick. Step back from the unit and visually check sampler alignment. A magnetic level can be placed on the assembly to check level.

9. Apply static weight and hammer percussion to advance the assembled outer casing until the drive head reaches the ground surface.

   NOTE: Activate hammer percussion whenever collecting soil. Percussion helps shear the soil at the leading end of the sampler so that it moves into the sample tube for increased recovery.

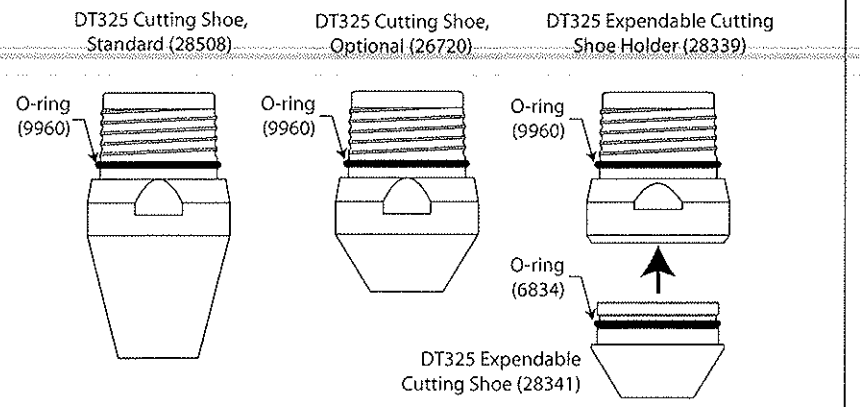


Figure 4.3
DT325 Cutting Shoes Utilize O-ring Seals

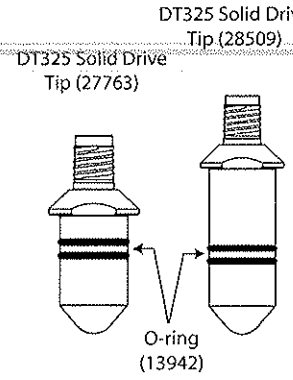


Figure 4.4
Solid Drive Points Utilize Two O-rings

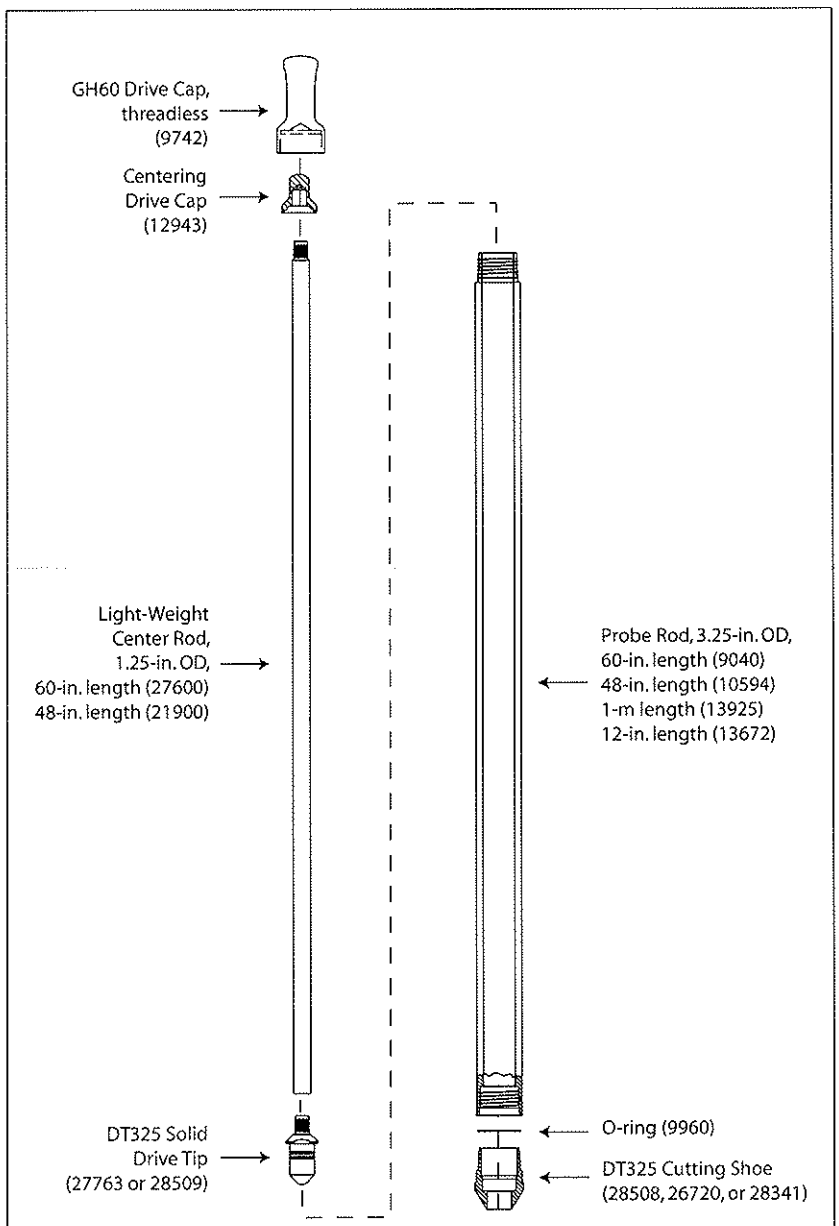


Figure 4.5
Assembly Using the DT325 Solid Drive Tip

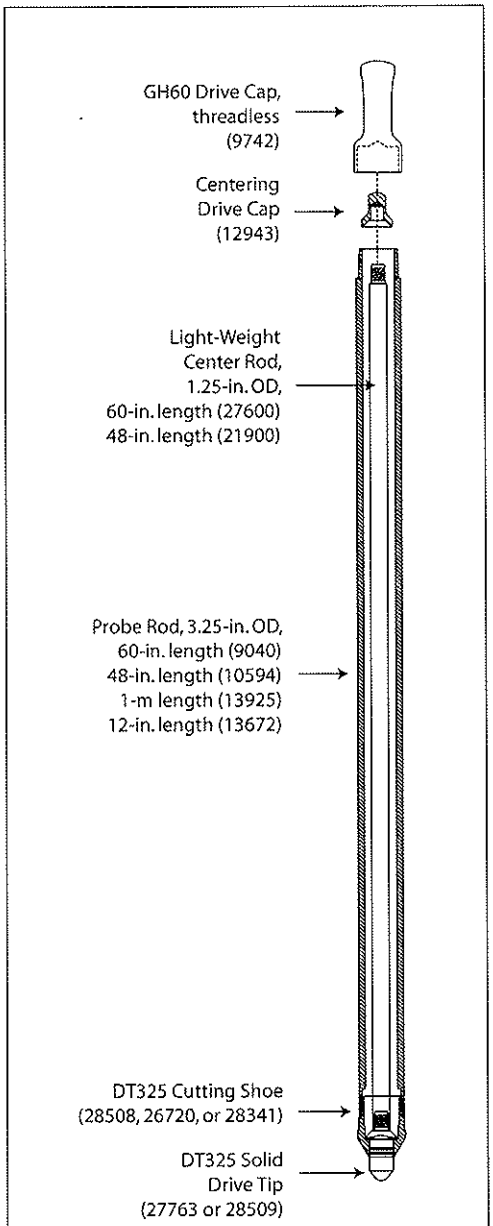


Figure 4.6
Completed Assembly

10. Raise the hammer assembly a few feet and retract the unit to provide access to the top of the outer casing assembly.

11. Remove the centering drive cap and 3.25-inch drive cap.

12. Add additional 1.25-inch light-weight center rods and 3.25-in. probe rods until the sampling interval is reached. At this point, the inner rods can be removed and an assembled sample sheath can be added (See Section 4.4)

4.4 Assembling the Sample Sheath

The sample sheath is used to support the weight of the 1.25-inch light-weight center rods and to protect the liner from damage while advancing the DT325 tool string. The process of assembling the sheath to collect soil samples is given below.

1. Place an O-ring onto the DT325 Liner Retainer. Note: No O-ring is needed for retainer 31039.
2. Slide the retainer ring onto the leading end of the liner. (Fig. 4.7).
3. Place the liner and retainer ring into either end of the sampler sheath (Fig. 4.8).
4. Thread the retainer ring onto the sample sheath. If the tools are clean, it should easily thread on easily by hand (Fig. 4.9).
5. On the opposite end of the sheath, thread on the DT325 Sheath Drive Head. The drive head will connect the sheath to the 1.25-inch light-weight center rods.

Figure 4.7. The retainer ring is placed on the end of the liner.

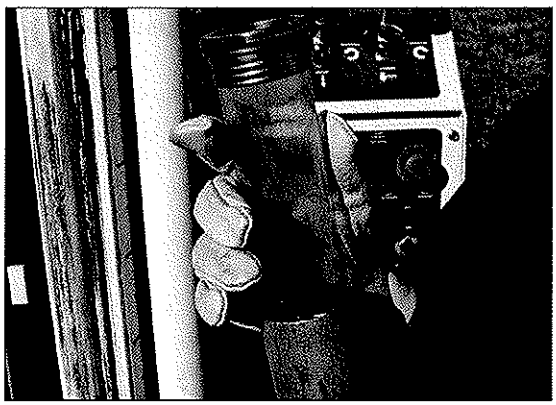


Figure 4.8. The liner and spacer ring are slid into the sample sheath.

Figure 4.9. Tighten the retainer ring by hand.

The sample sheath is now ready for soil core collection (Section 4.5).

4.5 Soil Core Collection

This section describes collection of continuous soil core samples from within the sealed outer casing of the DT325 Dual Tube Sampling System. The procedure is written for a sampling series that begins at the ground surface. Refer to Figure 4.10 for an illustration of the assembled sampler.

1. When using a DT325 Standard (28508) or Optional (26720) Cutting Shoe, install an O-ring (9960) at the base of the theads as shown in Figure 4.3. If using an expendable cutting shoe, install an O-ring (9960) on the DT325 Expendable Cutting Shoe Holder (28339) and one o-rings (6834) on the DT325 Expendable Cutting Shoe (28341).
2. Thread the DT325 Cutting Shoe or DT325 Expendable Point Holder onto the leading end of a 3.25-inch OD Probe Rod (Fig. 4.11). Completely tighten the cutting shoe or cutting shoe holder using a pipe wrench.
3. Insert the sample sheath assembly into the 3.25-inch OD probe rod.

4. Place a Centering Drive Cap (12943) on top of the DT325 Drive Head (Fig. 4.12) and a GH60 Threadless Drive Cap (9742) onto the 3.25-inch probe rod (outer casing, Fig. 4.13).

5. Raise the hydraulic hammer to its highest position by fully extending the probe cylinder.

6. Position the DT325 Sampler directly under the hammer with the cutting shoe centered between the toes of the probe foot (Fig. 4.14). The sampler should now be parallel to the probe derrick. Step back from the unit and visually check sampler alignment. A magnetic level can be placed on the assembly to check level.

7. Apply static weight and hammer percussion to advance the sampler unit until the drive head reaches the ground surface.

   NOTE: Activate hammer percussion whenever collecting soil. Percussion helps shear the soil at the leading end of the sampler so that it moves into the sample tube for increased recovery.

8. Raise the hammer assembly a few feet and retract the unit to provide access to the top of the sampler.

9. Remove the drive cap and thread an additional 1.25-inch light-weight center rod onto the center string. Place the adjustable rod clamp on the top of the 3.25-inch rods to keep the center rods from falling when they are removed (Fig. 4.15).

10. Pull up the 1.25-inch light-weight center rod string along with the sample tube (Fig. 4.16).

    To sample consecutive soil cores, advance a clean sample sheath and liner down the previously opened hole to the top of the next sampling interval. Add 1.25-inch light weight center rods as the sample sheath is lowered into the opened hole. An additional 1.25-inch light-weight center rod and 3.25-inch probe rod should be added. Drive the tool string the length of the sampler to collect the next soil core. Proceed to Section 4.6 for instructions on recovering the soil core from the sample sheath.

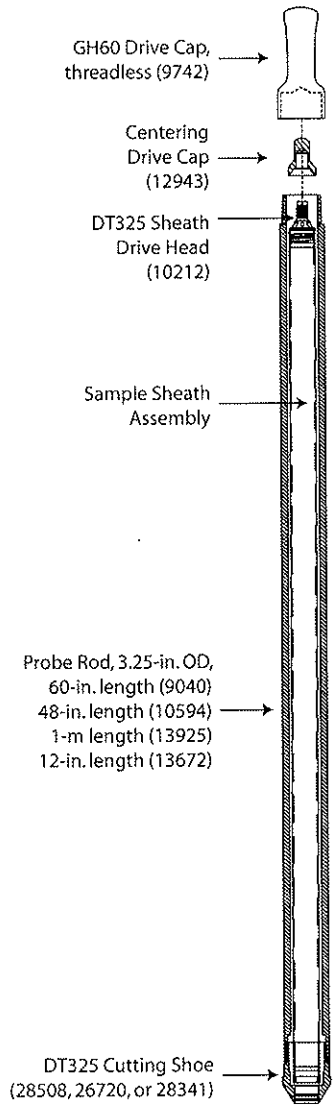


Figure 4.10
Assembled DT325 Sampler

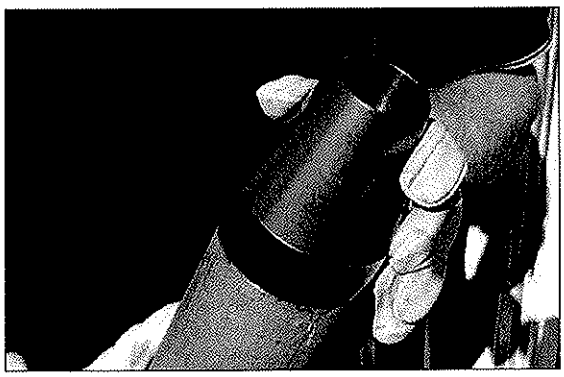


Figure 4.11. The cutting shoe is threaded onto the 3.25-inch probe rod.

Figure 4.12. A centering drive cap is placed on the DT325 Drive Head.

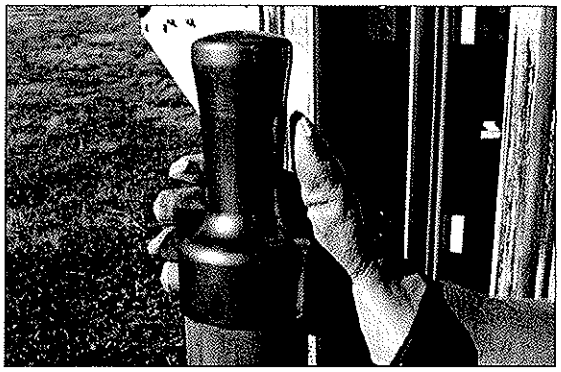


Figure 4.13. Place the threadless drive cap on the 3.25-inch probe rod.

Figure 4.14. The probe rod should be centered between the toes of the probe foot.

Figure 4.15. The adjustable rod clamp can be used when retrieving the sample.

Figure 4.16. The 1.25-inch light-weight center rods are pulled along with the sample tube.

4.6 Removing Filled Liner from the Sample Sheath

Place the sample tube into the vise. The liner retainer wrench can be used to remove the DT325 Liner Retainer and liner from the sample sheath. If possible, the retainer can be removed by hand (Fig. 4.17). The wrench can be used to gently knock off the retainer if necessary (Fig. 4.17). With the retainer removed, the liner and core can be withdrawn from the sample tube. A Hydraulic Liner Extruder is also available for mounting on your machine to remove liners (Fig. 4.19).

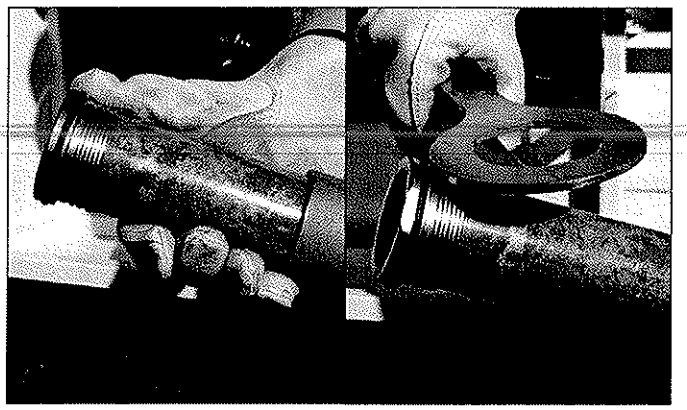


Figure 4.17. The retainer is removed from the sheath, either by hand or with the retainer wrench. Gently tap the retainer with the wrench to remove it from the liner.

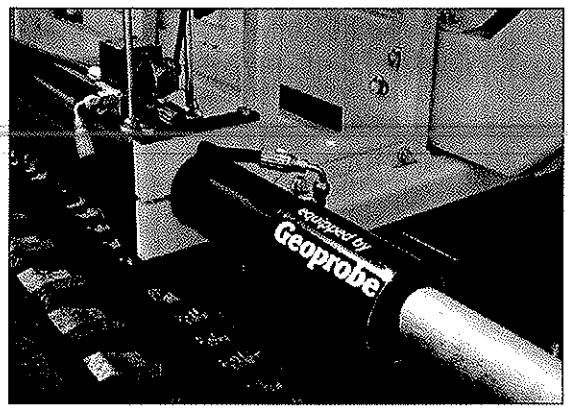


Figure 4.18. The Hydraulic Liner Extruder helps remove the liner.

4.7 Removing a Section of Liner with a DT325 Liner Cutter

The liner and core can be placed on the Universal Liner Holder. Use the DT325 Liner Cutter to safely expose the sample. Begin the cut at the opposite end of the core catcher (Fig. 4.19). It is a little thinner plastic, which makes it easier to begin the cut. Using both hands, smoothly pull the cutter through the liner. The slit liner can be removed and the core is exposed (Fig. 4.20).

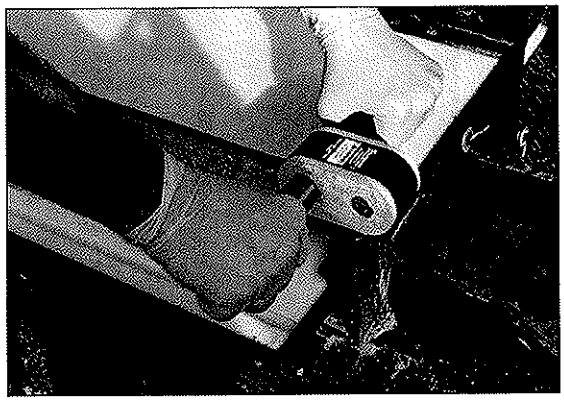


Figure 4.19. The DT325 Liner Cutter is used to safely make a longitudinal cut on the sample.

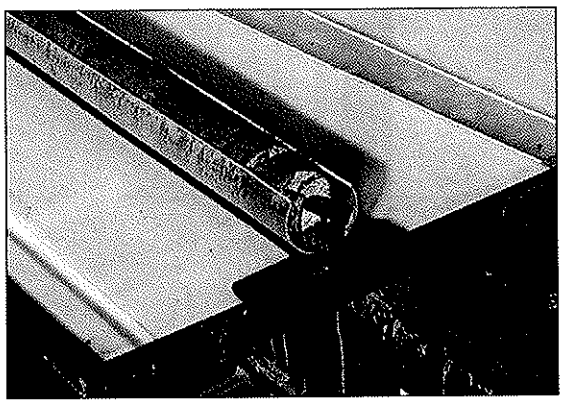


Figure 4.20. The core is exposed by the DT325 Liner Cutter.

4.8 Dual Tube Soil Sampling Tips

Saturated sands are the most difficult formations to sample with the DT325 system. Saturated conditions place positive pressure on the soil outside of the outer casing. When sampling in noncohesive formations (e.g. sands) below the water table, it may be necessary to add water to the outer casing to prevent formation heave. Adding water to the probe rods puts a positive head on the system and may keep formation material from flowing into the rods as the liner and soil sample are retracted. If a small amount of formation material is still drawn into the outer casing as the soil core is retrieved, the material may be displaced by slightly raising the outer casing while lowering the next new liner to depth. Water must be maintained within the outer casing during this process to overcome the hydraulic head imparted by the formation fluid. When retrieving, pull back the sample slowly.

DT325 core catcher liners will help considerably with sample recovery in non-cohesive soils and other materials that do not fill the liner diameter. Core catcher liners are not recommended for cohesive or expansive soils as the core catchers may actually inhibit soil movement into the liner. Also, using a shorter sample interval may improve sample recovery by minimizing wall friction as the material is sampled.

Certain soils have a tendency to exhibit plastic flow or extrusion characteristics. Allowing additional space for these materials will increase the speed of sampling because less time is spent cleaning overfilled sample sheaths. This will also yield a more representative sample. Using a sheath that is a foot or two longer than the sampling interval or using a shorter sample interval (under driving) can create a buffer zone. The DT325 Liner Spacer and Spacer Head were designed for these situations.

Some clay materials will extrude during sampling. Under these conditions, using a shorter sample interval (24-inch liners) may improve sample recovery by minimizing the wall friction as the material is sampled.

It is recommended that O-rings be used no the cutting shoe and liner retainer when sampling in clays. If they are not used, clay may build up between the sample sheath and the outer casing. It is not necessary to use o-rings in saturated sands and anytime water is present.

It may be helpful to mark the first 1.25-inch light-weight center rod attached to the sheath as an indicator that the sheath is next in line.

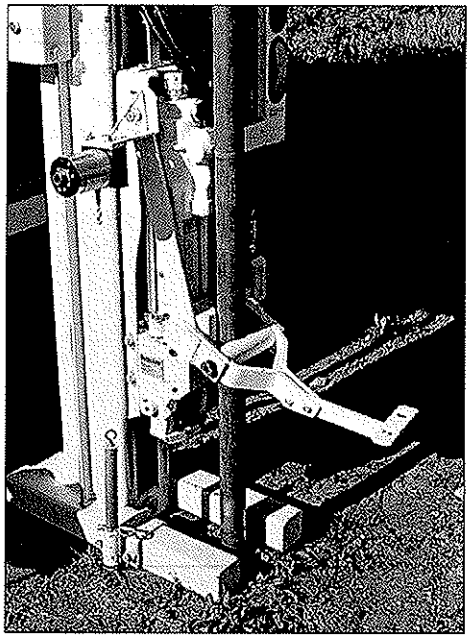


Figure 4.21. Outer casing may be retrieved with a pull cap or rod grip pull system if the probe hole is sealed with granular bentonite.

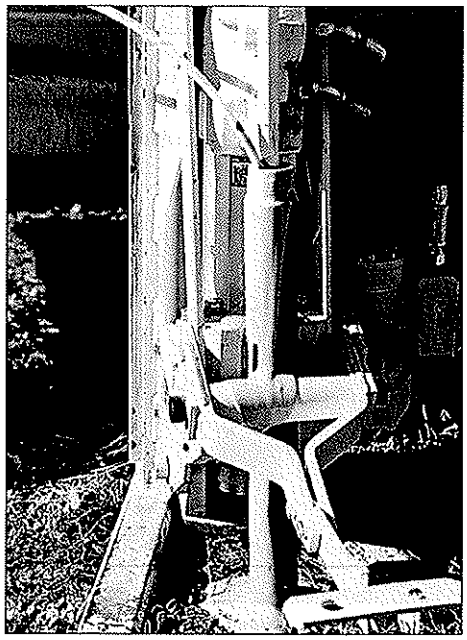


Figure 4.22. A grout machine and flexible tubing allow bottom-up grouting as the outer casing is retrieved.

4.9 Outer Casing Retrieval

The outer casing of the DT325 Dual Tube System may be retrieved in one of three ways:

1. Casing pulled then probe hole sealed from ground surface with granular bentonite.

   The outer casing may be pulled from the ground with the probe machine and a Pull Cap (13257) or a Rod Grip Pull System (for GH40 Hammers [12235] or for GH60 Hammers [9757]) if the probe hole is to be sealed with granular bentonite from the ground surface (Fig. 4.21). This method is used for shallow probe holes in stable formations only. Such conditions allow the entire probe hole to be sealed with granular bentonite.

2. Casing pulled with probe hole sealed from bottom-up during retrieval.

   Bottom-up grouting should be performed during casing retrieval in unstable formations where side slough is probable. Such conditions create void spaces in the probe hole if granular bentonite is installed from the ground surface.

   A GS500 or GS1000 Grout Machine is used to deliver a sealing material (high-solids bentonite slurry or neat cement grout) to the bottom of the outer casing through flexible tubing. The grout mix is pumped through the tubing to seal the void remaining as the outer casing is retrieved (Fig. 4.22). This is an advantage of the DT325 Dual Tube Sampling System as other soil samplers require a second set of tools to deliver grout to the bottom of the probe hole. Contact Geoprobe Systems® for more information on bottom-up grouting with the GS500 and GS1000 Grout Machines.

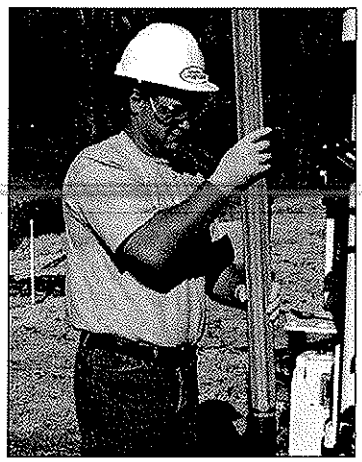


Figure 4.23. Geoprobe® prepacked screens may be installed through the outer casing when an expendable cutting shoe is used.

3. Casing pulled with Geoprobe Prepacked Screen Well installed during retrieval.

   The final option is to install a 2.5-inch OD Geoprobe® Prepacked Screen Monitoring Well in the probe hole during retrieval of the outer casing. A DT325 Expendable Cutting Shoe Holder (28339) and a DT325 Expendable Cutting Shoe (28341) allow the operator to collect continuous soil cores as the outer casing is driven to depth.

When sampling is complete, the outer rods are raised and the expendable cutting shoe is removed from the leading rod. This leaves an open casing through which a set of prepacked screens is lowered on the leading end of a PVC riser string. The well is finished, complete with grout barrier, bentonite well seal, and a high-solids bentonite slurry/neat cement grout, during retrieval of the outer casing.

Refer to Geoprobe® 0.5-in. x 1.4-in. OD and 0.75-in. x 1.4-in. OD Prepacked Screen Monitoring Wells Standard Operating Procedure (Geoprobe® Technical Bulletin No. 962000) for specific information on well installation.

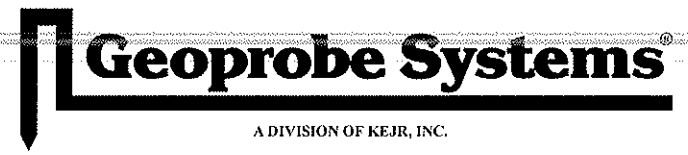
5.0 References

Geoprobe Systems®, 2003. *Tools Catalog, V. 6.*

Geoprobe Systems®, 2005. *Standard Operating Procedure. Geoprobe@ Pneumatic Slug Test Kit. Technical Bulletin No. 19344.*

Geoprobe Systems®, 2006. *Direct Push Installation of Devices for Active Soil Gas Sampling and Monitoring. Technical Bulletin No. MK3098.*

Geoprobe Systems®, 2006. *Standard Operating Procedure. 1.0-in. x 2.5-in. OD and 1.5-in. x 2.5-in. OD Prepacked Screen Monitoring Wells. Geoprobe® Technical Bulletin No. 962000.*



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Appendix C – Laboratory Standard Operating Procedures

List of Laboratory Standard Operating Procedures:

1. ST-IP-0013_Rev_25 Metals Water Digestion
2. ST-MT-0001_Rev_29 ICPMS Analysis
3. ST-RC-0020_Rev_22 Gross Alpha Beta
4. ST-RC-0025_Rev_19 Gamma Prep
5. ST-RC-0041_Rev_18 Ra 226-228
6. ST-RD-0102_Rev_19 Gamma Analysis
7. ST-RD-0403_Rev_20 GFPC
8. Dioxin and Furan in Soil by 8290A Prep_9488
9. Dioxin and Furan Water by 8290A Prep_12032
10. Dioxins and Furans in Water and Soil by 8290A_9476
11. Herbicides in Soil by 8151A Prep_10912
12. Herbicides in Soil by 8151A_9845
13. Herbicides in Water by 8151A Prep_10919
14. Herbicides in Water by 8151A_9202
15. ICP Metals in Water and Soil by 6010D_11931
16. ICP Metals in Water by 6010D Prep_8639
17. ICPMS Metals in Water & Soil by 6020A _11933
18. ICPMS Metals in Water by 6020A Prep_11937
19. Mercury in Soil by 7471B Prep_11948
20. Mercury in Water & Soil by 7470A & 7471B_7965
21. Mercury in Water by 7470A Prep_11924
22. Metals by ICP and ICPMS in Soil Prep_8636
23. PCBs in Soil by 8082A Prep_10927
24. PCBs in Soil by 8082B_10004
25. PCBs in Water by 8082A Prep_10920
26. PCBs in Water by 8082A_9238
27. Pesticides in Soil by 8081B Prep_10926
28. Pesticides in Soil By 8081B_9232
29. Pesticides in Water by 8081B Prep_10920
30. Pesticides in Water by 8081B_9999
31. SVOCs in Soil by 8270D and 8270D SIM Prep_10928
32. SVOCs in Water & Soil by 8270D SIM_9995
33. SVOCs in Water & Soil by 8270D_9617
34. SVOCs in Water by 8270D Prep_11432
35. SVOCs in Water by 8270D SIM Prep_10931
36. VOCs in Soil by 8260C_8236
37. VOCs in Soil Prep by 5035A_11242
38. VOCs in Water by 8260C_8194

Title: ACID DIGESTION OF AQUEOUS SAMPLES FOR METALS [SW-846 3005A, 3010A; EPA 200.7, 200.8]

Approvals (Signature/Date):

Cory Buffington — 10/31/19
Cory Buffington — Date
Department Manager

Michael Ridenhower — 10/31/19
Michael Ridenhower — Date
Health & Safety Manager / Coordinator

Kristen Ely — 10/31/19
Kristen Ely — Date
Quality Assurance Manager

Jodie Carnes — 10/30/19
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Laboratory Director

This SOP was previously identified as SOP No. ST-IP-0013 Rev. 24

Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure is applicable to the preparation of digestates from aqueous samples, including SPLP, CWET and TCLP extracts, and wastes that contain suspended solids, for subsequent analysis by ICP (Inductively Coupled Argon Plasma Spectroscopy) or ICP/MS (Inductively Coupled Argon Plasma Mass Spectroscopy) for total recoverable metals.

1.2 This SOP is based on SW846 Method 3005A, 3010A, EPA Method 200.7 and EPA Method 200.8.

1.3 Additional metals may be processed by this method, assuming that performance criteria of the determinative method are met.

1.4 It is mandatory that Clean Water Act (CWA) samples be digested per this procedure prior to analysis.

1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Water samples are refluxed with nitric acid in a hot block vessel until the digestate is light in color. The digestate is reduced to a low volume and further refluxed with hydrochloric acid. The cooled digestate is brought to a known volume for analysis by ICP or ICP/MS.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Laboratory Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Total Recoverable Metals: The concentration of target metals determined on an unfiltered sample after vigorous acid digestion. (samples may be filtered AFTER digestion)

3.3 Dissolved Metals: Those elements which pass through a 0.45 µm membrane filter (Sample is filtered BEFORE acidification and digestion)

4.0 INTERFERENCES

4.1 Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc). Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3 PRIMARY MATERIALS USED
The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm- (TWA) 4 ppm- (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm- (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Hot block or other heating source capable of maintaining a temperature of 90 to 95 °C.

6.2 Laboratory fume hood.

6.3 Thermometer, temperature range of 0 – 200 °C

6.4 Adjustable pipettes (0.1-1mL)

6.5 Adjustable Reagent Dispensers

6.6 50-mL hot block digestion vessels (Class A)

6.7 Watch glasses

6.8 Vacuum pump

6.9 Vacuum manifold

6.10 0.45 µm vacuum filter units (with plugs)

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI water: Obtained by the Milli-Q System (resistivity ≥1 Mohm-cm)

7.3 Nitric acid, concentrated, trace metal grade or better

7.4 Hydrochloric acid, concentrated, trace metal grade or better

7.5 LCS standard, NIST traceable

7.6 Matrix Spike standard, NIST traceable

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Aqueous samples must be acidified at the time of collection to a pH of 2 or less with nitric acid.

8.2 If samples are preserved by the lab, the samples must sit 24 hours before digestion.

8.3 Sample containers may be either plastic or glass, and must be pre-cleaned by vendor.

8.4 Sample digestion and analysis must be completed within six months (180 days) of sample collection.

8.5 Leachates must be digested and analyzed within six months (180 days) of the beginning of the leaching.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. When there is no preparation method the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument/equipment conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and a Matrix Spike/ Matrix Spike Duplicate (MS/MSD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Sample Duplicate (DU) may be performed upon client request, and are noted in LIMS.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.2.1 For Water analyses, the method blank is comprised of a 2% HNO_3 solution.

9.3 **Laboratory Control Sample**

9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.2.1 For Water analyses, the LCS is comprised of a 2% HNO_3 solution, fortified with target analytes.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 For **6010B, 6010C, 6010D, 6020**, **6020A and 6020B**:

9.4.3.1 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.4.4 For **200.7** and **200.8**:

9.4.4.1 MS/MSD are performed every 10 samples or per batch whichever is shorter.

9.4.5 If there is insufficient sample to perform a MS/MSD, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume.

9.5 **Sample Duplicate (DU)**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.5.3 A Sample Duplicate is only performed when client requested.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Hot block must be checked daily when in use.

10.1.1 Temperature is documented in the LIMS.

10.1.2 A calibrated thermometer is suspended in sand in a digestion vessel of water and brought to the proper temperature.

10.1.2.1 See SOP ST-QA-0005 for information regarding calibration of thermometers.

10.2 Instrument calibration is discussed in the respective analytical SOPs: ST-MT-0001 (ICP/MS), and ST-MT-0003 (ICP).
10.3 Balance must be checked daily when in use.
10.3.1 Balance calibration is discussed in the respective analytical SOP: ST-QA-0005
10.4 Pipettes must be checked daily when in use.
10.4.1 Pipette calibration is discussed in the respective analytical SOP; ST-QA-0005

11.0 PROCEDURE

11.1 For Dissolved metals, the sample must be filtered BEFORE preservation
11.1.1 See SOP ST-IP-0015 (Filtration Procedure)

11.2 For Total Recoverable metals, the sample must be preserved before digestion. See section 8.0

11.3 For Acid Leach: See Attachment 1
11.3.1 This does NOT include TCLP, CWET, SPLP or Kd Leach

11.4 For Suspended Sample (digested by radiochemistry) See Attachment 2

11.5 For TCLP, CWET and SPLP received the day after the batch is completed, containers with a label saying "unpreserved" and samples with particles around spout of container, check preservation before analysis. If pH is <2, add HNO_3 as needed
11.5.1 After preservation, wait 24 hours before digestion
11.5.2 If the amount of acid added is more than 1% of the samples total volume, a dilution factor must be added in. See Attachment 3

11.6 Label digestion vessel with sample ID or QC identifier.

11.7 Transfer 50 mL of well-mixed sample to a digestion vessel.
11.7.1 If a smaller aliquots of sample is used the applicable reagents are reduced accordingly. Alternatively the sample may require dilutions to reduce interferences. If the sample is diluted during the digestion bring total volume to 50 mL with DI water prior to adding the reagents.
11.7.2 For the MB and LCS, use 50 mL of 2% HNO_3 solution.
11.7.3 Spike LCS and MS/MSD with spiking mix applicable to the requested analysis.
11.7.4 Document spiking volumes and standard numbers in LIMS

11.8 When preparing TCLP leachate samples, aliquot 20 mL of leachate and dilute to 50 mL with DI, unless client project requirements specify no dilution.
11.8.1 Note: this dilution applies to all QC samples.

11.9 When preparing CWET samples, aliquot 10 mL of leachate and dilute to 50 mL with DI, unless client project requirements specify no dilution.
11.9.1 Note: this dilution applies to all QC samples.

11.10 When preparing SPLPE & SPLPW transfer 50 mL of well mixed sample to a digestion vessel

11.11 **Digestion – SW-846 3010A**

NOTE: this digestion procedure is also used for 200.7 and 200.8 Non-Potable Waters

11.11.1 Add 2.5 mL of concentrated nitric acid.
11.11.1.1 If the sample changes color with the addition of nitric acid, repeat the addition of acid and refluxing until digestion is complete, which is indicated by

the appearance of a light colored liquid which does not change with continue refluxing.

11.11.2 Place watch glasses on top of all digestion vessels.

11.11.3 Place the digestion vessel in a hot block at 90 °C – 95 °C and evaporate, without boiling, for approximately 10 to 12 hrs.

11.11.3.1 Note: do not allow sample to go to dryness, as loss of target metals may result.

11.11.4 Cool vessel.

11.11.5 Add 5.0 mL of 1:1 HCl to the digestate, and place back in hot block for an additional 15 minutes.

11.11.6 Cool the samples; wash down the vessel wall with DI water to dissolve any precipitation to a final volume 50 mL.

11.11.7 Cap and mix sample thoroughly and allow too settle.

11.11.8 After samples are brought to 50mL, filtration may be needed for some samples (such as those with large amounts of sediment) before analysis

11.11.8.1 NOTE: The MB and LCS must also be filtered

11.11.8.2 Label clean digestion tubes

11.11.8.3 Attach filter to the sample digestion tube. Attach the clean, labeled digestion tube to the other side of the filter.

11.11.8.4 Attach vacuum manifold to the vacuum pump, turn on the pump.

11.11.8.5 Either use finger or place plug over hole in filter

11.11.8.6 Invert, and attach to the vacuum manifold

11.11.8.6.1 If the sample is difficult to filter using a digestion tube filter, a vacuum pump filter may be used to filter out remaining sediment. The MB and LCS must also be filtered.

11.11.8.7 Once all liquid has gone through, turn off the vacuum and remove the sample.

11.11.8.8 Cap the sample

11.11.9 The sample is now ready for analysis via the appropriate analytical SOP. Store digestates in designated cabinet, and transfer the load sheet to the analyst.

11.12 **Digestion - SW-846 3005A**

11.12.1 Add 1.0mL of concentrated HNO3 and 2.5mL of concentrated HCl to each sample. Place a watch glass on top of all digestion vessels. Gently heat the block digestion vial to a temperature 90-95°C until the volume is about 7.5mL-10mL, approximately 18 to 20 hours. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

11.12.1.1 NOTE: IF a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

11.12.2 Wash down the inside of the digestion vessel with DI water. Dilute the sample digestate to 50mL with DI water.

11.12.3 The sample is now ready for analysis via the appropriate analytical SOP. Store digestates in designated cabinet, and transfer the load sheet to the analyst.

11.13 **Digestion -EPA 200.8 Drinking Water**

11.13.1 Add 1.0mL of 1:1 HNO_3 and 0.5mL of 1:1 HCl to each sample. Gently heat the block digestion vial to a temperature 90-95C and reduced the sample volume to about 10mL without boiling, approximatley 7 hours. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

11.13.1.1 NOTE: If a volume sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

11.13.2 Cover the digestion tube with a disposable watch glass and reflux the sample for 30 minutes.

11.13.3 Wash down the inside of the block digestion vial with DI water. Dilute the sample digestate to 50mL with DI water.

11.13.4 The sample is now ready for analysis by ICP/MS. Store digestates in designated cabinet, and transfer the load sheet to the analyst.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the ST-QAM.

12.2 Specific calculations are included in the respective analytical SOPs: ST-MT-0001 (ICP/MS), and ST-MT-0003 (ICP).

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment, acceptance criteria and corrective actions are included in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

13.2 Data assessment does not pertain to this sample preparation procedure.

13.3 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036.

14.0 METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM .

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A"

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 SW-846 Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy", Revision 1, July 1992

17.2 SW-846 Method 3005A , "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy", Revision 1, July 1992

17.3 EPA Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry," Revision 4.4.

17.4 EPA Method 200.8, "Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry", Revision 5.4.

17.5 TestAmerica St. Louis Laboratory Quality Assurance Manual (ST-QAM), current revision

17.6 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.7 Associated SOPs, current revisions:

17.7.1 ST-PM-0002, Sample Receipt and Chain of Custody
17.7.2 CA-Q-S-006, Detection and Quantitation Limits
17.7.3 ST-QA-0002, Standard and Reagent Preparation
17.7.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes

17.7.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.7.6 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.7.7 ST-IP-0004, Labware Preparation for Inorganic and Trace Metal Analysis
17.7.8 ST-IP-0015, Filtration Procedure
17.7.9 ST-MT-0001, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry
17.7.10 ST-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method for Trace Element Analysis

18.0 MODIFICATIONS TO THE REFERENCED METHODMETHOD

18.1 **3010A**

18.1.1 TestAmerica St. Louis uses a 50 mL sample aliquot size instead of the 100 mL aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.
18.1.2 Additionally, sample reagent volumes needed for digestion have been reduced to reflect the reduction in sample volume.
18.1.3 Acid concentrations have been modified to optimize recoveries and to combine prep methods. The final acid concentration in the digestate is 5% HCl and 5% HNO_3 (v/v%).

18.2 **3005A**

18.2.1 TestAmerica St. Louis uses a 50 mL sample aliquot size instead of the 100 mL aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.
18.2.2 Additionally, sample reagent volumes needed for digestion have been reduced to reflect the reduction in sample volume.

18.3 **200.8 (Drinking Water)**

18.3.1 TestAmerica St. Louis uses a 50 mL sample aliquot size instead of the 100 mL aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.
18.3.2 Additionally, sample reagent volumes needed for digestion have been reduced to reflect the reduction in sample volume.
18.3.3 The method allows direct analysis, if the sample is a drinking water, and the turbidity of the sample is < 1NTU. TestAmerica St. Louis digests all samples, unless requested otherwise by the client
18.3.4 The method specifies the use of a hot plate, and that an uncovered Griffin beaker containing 50mL of water should be maintained at a temperature approximately to, but no higher than 85°C. But once the beaker is covered with a watch glass the temperature of the water will rise to approximately 95°C. TestAmerica St. Louis measures the temperature in a closed container, and so uses a range of 90-95°C.

18.4 **200.7, 200.8 (Non Potable Water)**

18.4.1 TestAmerica St. Louis uses a 50 mL sample aliquot size instead of the 100 mL aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.
18.4.2 Additionally, sample reagent volumes needed for digestion have been reduced to reflect the reduction in sample volume.
18.4.3 The method allows direct analysis, if the sample is a drinking water, and the turbidity of the sample is < 1NTU. TestAmerica St. Louis digests all samples, unless requested otherwise by the client
18.4.4 The method specifies the use of a hot plate, and that an uncovered Griffin beaker containing 50mL of water should be maintained at a temperature approximately to, but

no higher than 85°C. But once the beaker is covered with a watch glass the temperature of the water will rise to approximately 95°C. TestAmerica St. Louis measures the temperature in a closed container, and so uses a range of 90-95°C.

18.4.5 Acid concentrations have been modified to optimize recoveries and to combine prep methods. The final acid concentration in the digestate is 5% HCl and 5% HNO_3 (v/v%):

19.0 CHANGES TO PREVIOUS REVISION

19.1 Removed Plunger Filter from section 6.0.
19.2 Up dated section 11.10 regarding the use of plunger filters.
19.3 Rev 17:
- 19.3.1 Updated digestion time for sample to evaporate in section 11.7.
- 19.3.2 Updated where spiking volumes records to be stored in section 11.2.4.
- 19.3.3 Updated pipette and reagent dispenser range used in section 6.0.

19.4 Revision 18:
- 19.4.1 Updated matrix for method blank and LCS for Au, Ta and Pd in section 9 and 11

19.5 Revision 19:
- 19.5.1 Corrected digestate cleaning method in section 8.2
- 19.5.2 Removed reference to spiking concentrations in section 11.5.
- 19.5.3 Updated section 15.1
- 19.5.4 Updated QAM reference in Section 14.

19.6 Revision 20
- 19.6.1 Changed starting matrix for MB and LCS, from DI to 2% HNO_3
- 19.6.2 Changed final acid concentration, from 3% HNO_3/2%HCl, to 5%HNO_3/5%HCl
- 19.6.3 Added modifications to section 18, clarified which method was being modified.
- 19.6.4 Removed Table 1 (DOE low level spike)- not reference in SOP, levels can be found in LIMS.
- 19.6.5 Added reference to dissolved metals
- 19.6.6 Added reference to filtration procedure

19.7 Revision 21 (08/28/2015)
- 19.7.1 Grammatical corrections throughout
- 19.7.2 Section 11: removed reference to low level TCLP digestion.

19.8 Rev. 22 (08/12/2016)
- 19.8.1 Made clarifications throughout
- 19.8.2 added vacuum manifold to supplies
- 19.8.3 added use of watch glasses to Section 11
- 19.8.4 added filtration procedure for samples that have already been digested
- 19.8.5 added reference to methods 6010D and 6020B

19.9 Rev. 23 (08/10/17)
- 19.9.1 Annual review. Grammatical/punctuation corrections

19.10 Rev. 24 (10/31/2018) Technical Review A. Mazariegos and F. Cruz, QA Review – K. Ely
- 19.10.1 Added references to method 3005A and 200.8 for drinking water throughout
- 19.10.2 Section 7: corrected DI resistivity requirement
- 19.10.3 Section 10: added balance and piptte verification requirement
- 19.10.4 Section 11
  - 19.10.4.1 Section 11.5: updated when necessary to check sample preservation
  - 19.10.4.2 Section 11.7: removed reference to Type 1 DI
  - 19.10.4.3 Section 11.11: Noted that this digestion procedure was for 3010A, and also used for 200.7 and 200.8 non-potable water
    - 19.10.4.3.1 Added a second option for filtering
  - 19.10.4.4 Section 11.12: Added digestion for method 3005A
  - 19.10.4.5 Section 11.13: Added digestion for method 200.8 for drinking water

19.10.5 Section 17: added reference to corporate SOP CA-Q-S-006, removed retired local SOP ST-QA-0016
19.10.6 Section 18: updated modification section, added modifications/clarifications for 3005A and 200.8 DW

19.11 Rev. 25 (11/1/2019)
19.11.1 Added Eurofins logo, updated copyright information.
19.11.2 Changed “Total Metals” to “Total Recoverable Metals” throughout.
19.11.3 Section 9: removed duplicated verbiage about non-conformances.
19.11.4 Section 11: made minor clarifications throughout.

Attachment 1
Acid Leach

1- Samples are leached by pre-prep. A spreadsheet is made
   a. QA_Public\FORMS\Rad\Rad-0085_Sample_Leach_Log

2- In Spreadsheet:
   a. Complete Fields (use info from Acid Leach batch)
      i. Sample ID
      ii. Sample Weight
      iii. Leach Volume
   b. For Soils
      i. We want the Gram Equivalent (column F) to equal approx 0.5g. Enter various numbers into mL's (column E) until this happens.
   c. Save and print spreadsheet (this will go with batch papers)

3- In TALS
   a. Create batch, enter sample ID's
   b. For INITIAL volume, enter Gram Equivalent number from spreadsheet (column F)
   c. For FINAL volume, enter 50mL
   d. To prep:
      i. Pipette amount from mL's (column E) into digestion tube
      ii. Bring up to a final volume of 50mL

4- Fill out rest of batch information and digest as normal, using water digestion.

Attachment 2
Suspended Solids (Prepped by Radiochemistry)

1- Samples are digested by Rad. A spreadsheet is made
    a. QA_Public\Forms\Rad\RAD-0082_Suspended_Fraction_Calcs
2- In Spreadsheet:
    a. Complete Fields for
        i. Enter Test
        ii. Enter Batch #
        iii. Sample ID
    b. For Waters
        i. We want column O to equal approx 50mL. Enter various numbers into column J until this happens
    c. For Soils
        i. We want column P to equal approx 0.5g. Enter various numbers into column I until this happens.
    d. Save and print spreadsheet (this will go with batch papers)
3- In TALS
    a. Create batch
    b. In "Batch Information", enter information for
        i. Digestion tube lot
        ii. Pipette
        iii. Comment (sample digested in Rad)
    c. Write an NCM

        Prep Batch XXXXX Suspended

        The digestate is the suspended portion of the sample. The suspended portion was digested by the radiochemistry department. The MB/LCS will be prepped post-digestion

    d. For INITIAL volume, enter number from spreadsheet (column O or P)
    e. For FINAL volume, enter 50mL
    f. To prep:
        i. Pipette initial volume of the sample into digestion tube
        ii. Bring up to a final volume of 50mL
            1. ICP- use 5% HCl / 5% HNO_3 as diluent
            2. ICPMS- use 2% HCl /2% HNO_3 as diluent
4- Give analyst samples and paperwork

Attachment 3

Determining Dilution Factor

A dilution factor is needed if the amount of nitric acid being added is > 1% of the samples total volume.

- To find the samples total volume use a graduated cylinder and subtract how much acid was used to preserve it.
- If a larger container was used to preserve the sample, add all of the sample back into the original bottle. Wait 24 hours before digestion.

How to use the dilution factor spreadsheet

1. Use form INORG-0165_Dilution_Factor
2. Divide the aliquot taken of the sample by the dilution factor to get the adjusted aliquot
3. In TALS write the adjusted aliquot in place of the initial amount.
4. Add the dilution factor Doc to your prep batch and narrate using the NCM below (editing the bracketed items.)

- NCM -
<PrepAnalyticalBatch>
Due to the samples elevated pH, a dilution factor was used after exceeding the maximum amount of acid allowed for preservation. Dilutions were done as follows:
The attached spreadsheet calculates the needed dilution for the initial and the final volume of the sample. We took our initial aliquot (0.5mL) and divided it by the dilution factor (1.0196), to find our adjusted aliquot (0.4904). Regulatory documents require a 24-hour waiting period from the time of the addition of the acid preservative to the time of digestion. **<&commamerge&>**

Example:

Analyst added 5mL of HNO_3 to a sample. When the preserved sample was poured into a graduated cylinder, it measured at 210mL.

210mL(total) – 5mL(acid) = 205mL (sample)

Date: date here
Analyst: analyst name here

Sample	Initial Volume (mL)	Final Volume (mL)	Dilution
XXXXXX	205	210	1.02439

Analyst then pours up 50mL of the sample

In TALS, put 48.8mL as the initial volume (50mL / 1.02439 = 48.8mL)

Attach spreadsheet to the prep batch, and write an NCM

Title: ANALYSIS OF METALS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY [SW-846 6020; SW-846 6020A; SW-846 6020B; EPA 200.8]

Approvals (Signature/Date):

Cory Buffington — 10/31/19
Department Manager — Date

Michael Ridenhower — 10/31/19
Health & Safety Manager / Coordinator — Date

Kristen Ely — 10/31/19
Quality Assurance Manager — Date

Jodie Carnes — 10/30/19
Laboratory Director — Date

This SOP was previously identified as SOP No. ST-MT-0001 Rev. 28

Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA SW846 Method 6020, 6020A , 6020B and EPA 200.8 .

1.2. This method is applicable to surface, and saline waters; soil and waste samples.

1.3. The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples for Metals (SW-846 3005A, 3010A, EPA 200.7 and EPA 200.8) and the soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils (SW846 Method 3050B).

1.4. The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

1.4.1. Additional elements may be amendable to this method provided the laboratory has established a MDL and the elements meets the QC requirements as prescribed in the associated preparation and analysis SOP.

2.0 SUMMARY OF METHOD

2.1. Sample digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio frequency plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3.0 DEFINITIONS

3.1. See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2. MDL: Method Detection Limit

3.3. EPA and SW-846 methodology use different terminology. Our SOP references the SW 846 terminology:

3.3.1. The ICV satisfies the QCS requirements found in method 200.8 .

3.3.2. The LCS satisfies the requirements of the LFB found in method 200.8.

3.3.3. The MS satisfies the requirements of the LFM found in method 200.8.

3.3.4. The MB satisfies the requirements of the LRB found in method 200.8.

3.4. Other terminology equivalents (used in SOP or LIMS)

3.4.1. ICSA and ICSAB = SIC

3.4.2. RL = LLOQ

3.4.3. LLICV = LLC = CRI

3.4.4. Dilution test = serial dilution

3.5. Dissolved Metals: Those elements which pass through a 0.45 µm membrane filter (Sample is filtered BEFORE preservation)

3.6. Suspended Metals: Those elements retained by a 0.45 µm filter

3.7. Total Recoverable Metals: The concentration determined on an unfiltered sample following vigorous digestion

4.0 INTERFERENCES

4.1. Isobaric elemental interferences: Isobaric elemental interferences associated with naturally occurring isotopes

are automatically corrected by the instrument software.

4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences. This may be done with either interference equations or collision cell technology.

4.3. Common molecular ion interferences are listed in Table 3 of this SOP.

4.4. Matrix interferences: Internal standards are used to correct for some matrix interferences.

4.4.1. Internal standards are added at a level to give approximately 100,000 - 10,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ± 50 amu of the mass of the affected analyte.

4.4.2. Severe matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the initial calibration blank.

5.0 SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1. The ICP plasma emits strong UV light, harmful to vision. Analysts must avoid looking directly at the plasma.

5.3. PRIMARY MATERIALS USED

5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1. Agilent 7500/ Agilent 7700, each includes the following:
- 6.1.1. Auto sampler
- 6.1.2. Chiller (water cooling device)
- 6.1.3. Peristaltic pump
- 6.1.4. Vacuum pump

6.2. Helium gas: 5.5 trace analytical grade

6.3. Argon gas: High-purity grade (99.99%)

6.4. Calibrated automatic pipettes

6.5. Teflon® flasks

6.6. Instrument software: Mass Hunter

- 6.6.1. Software versions are documented in the instrument maintenance log.

7.0 REAGENTS AND STANDARD

7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2. Concentrated nitric acid (HNO_3), trace metal grade

7.3. Concentrated hydrochloric acid (HCl), trace metal grade

7.4. DI water from the Millipore unit
- 7.4.1. Water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Water must be shown to have a resistivity greater than 1 Mohm-cm.

7.5. 2% HCl / 2% HNO_3
- 7.5.1. Fill a 20L carboy up approximately halfway with DI water
- 7.5.2. Add 400 mL of HCl and 400 mL of HNO_3.
- 7.5.3. Fill the carboy up to the 20L line, cap tightly, then shake to mix.

7.6. Standards, NIST traceable (or equivalent)
- 7.6.1. Purchased as custom multi-element mixes or as single-element solutions
- 7.6.2. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.
- 7.6.3. Working standards may be used for up to 6 months, with the exception of the ICV, ICSA, ICSAB
  - 7.6.3.1. The ICV must be prepared daily, and is used to verify that the calibration standards are still viable.
  - 7.6.3.2. For 6020B the ICSA and ICSAB must be prepared weekly
- 7.6.4. Standards should be prepared in a matrix of 2% hydrochloric and 2% nitric acid.
- 7.6.5. See either LIMS, ST-MT-WI-0001 (standard analytes) or ST-MT-WI-0003 (ISO-U) on how to make ICPMS standards.

7.6.6. Internal Standard Solution: Prepare internal standards (Li6, Sc, Ge, In, Ho, Ir) when needed.
7.6.6.1. The ID for the internal standard must be written on the run log daily.
7.6.7. Tuning solution: Prepare tuning solution (Be, Ba, Ce, Co, In, Pb, Li, Mg, Rh, Tl, Y) when needed.
7.6.7.1. The ID for the tuning solution must be written on the run log daily.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2. Aqueous samples must be digested before analysis using an appropriate digestion procedure (see ST-IP-0013).

8.3. Soil or waste samples are digested before analysis using an appropriate digestion procedure (see ST-IP-0002).

8.4. Digestate holding time is 6 months from sample collection.

9.0 QUALITY CONTROL

9.1. **Batch**
9.1.1. A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.
9.1.3. For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD an LCS Duplicate (LCSD) is prepared and analyzed.

9.2. **Method Blank (MB)**
9.2.1. A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
9.2.2. A method blank must be prepared with every sample batch.
9.2.3. For water batches, 2% HNO_3 is used.
9.2.4. For soil batches, glass beads or Teflon chips are used.
9.2.5. See Section 13 or LIMS for acceptance criteria.

9.3. **Laboratory Control Sample (LCS)**
9.3.1. An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.3.2. An LCS must be prepared with every sample batch.
9.3.3. For water batches, 2% HNO_3 spiked with the analytes of interest is used
9.3.4. For soil batches, a commercially available solid reference material (SRM) is used. Where an SRM is not available, or the manufacturer limits are not sufficient for client required limits, glass beads or Teflon chips spiked with an aqueous solution of the analytes of interest is used
9.3.5. See Section 13 or LIMS for acceptance criteria.

9.4. **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**
9.4.1. A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.4.2. See Section 13 or LIMS for acceptance criteria.

9.5. **Dilution Test (SD)**
9.5.1. A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.

9.5.2. The test is performed by running a sample at a 5x (1:4) dilution.
9.5.3. Samples identified as field blanks cannot be used for dilution tests.
9.5.4. See Section 13 or LIMS for acceptance criteria.

9.6. **Post Digestion Spike** (PDS)
9.6.1. A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process, with a spike concentration between 10-100 times the MDL (unless specific project/program criteria is given)
9.6.2. **200.8**: A PDS is not applicable for this method
9.6.3. **6020 and 6020B**: A PDS is analyzed with every batch
9.6.4. **6020A**: The method stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a dilution test on the sample. If the PDS is acceptable, the laboratory is not required to perform a dilution test. Since the laboratory has elected to perform the dilution test routinely, the intermediate step of a post digestion spike is not performed.
9.6.4.1. For client project or programs requiring a PDS, the laboratory will include a PDS in the batch in addition to the dilution test. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.
9.6.5. See Section 13 or LIMS for acceptance criteria.

9.7. **Method of Standard Addition** (MSA)
9.7.1. **6020/6020A/6020B**
9.7.2. This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.
9.7.3. MSA are not required by methods 6020, 6020A or 6020B.
9.7.4. MSA are not considered standard batch QC and if required by the client, must appear in the sample comment section in LIMS.
9.7.5. MSA is required by SW846 Method 1311 when the MS/MSD recovery is less than 50%, analyte concentration is less than and within 20% of its regulatory limit.
9.7.6. See Appendix I for more information on MSA

9.8. **Procedural Variations/ Nonconformance and Corrective Action**
9.8.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

Follow the instrument start-up procedure outlined in the manufacturers operating manual.
10.1. Allow the instrument to warm up for at least 30 minutes.
10.2. Cone Conditioning
10.2.1. Aspirating an ICSA solution for at least 1 hour can enhance instrument performance. This procedure should be used after a thorough cleaning of the interface cones or the installation of new cones takes place.

10.3. Rinse Time Determination
10.3.1. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in this SOP it can be demonstrated that a shorter rinse time may be used.
10.3.1.1. To determine the appropriate rinse time, a linear range verification standard should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for the system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an

excessive rinse time would be required at the linear range level). The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

10.4. Instrument Tuning

10.4.1. Frequency:

10.4.1.1. Daily with each initial calibration

10.4.2. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Li, Y, Tl, Co, In, and Ce.

10.4.3. Tune Criteria:

10.4.3.1. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.4.3.1.1. Resolution

10.4.3.1.1.1 6020/6020A/6020B: peak width must be < 0.9 amu at 10% peak height

10.4.3.1.1.2 200.8: peak width of approximately 0.75 amu at 5% peak height

10.4.3.1.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.

10.4.3.1.3. The tuning elements must have RSD below 5%. Doubly-charged ions and oxides must be below 3.0%.

10.4.3.1.4. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.5. **Initial Calibration**

10.5.1. Multi-point Calibration:

10.5.1.1. A calibration curve, consisting of 3 standards and a blank, must be analyzed daily.

10.5.1.2. Calibration criteria:

10.5.1.2.1. **200.8**: No criteria

10.5.1.2.2. **6020/6020A**: Correlation Coefficient of ≥ 0.998

10.5.1.2.3. **6020B**: Correlation Coefficient of ≥ 0.995

10.5.1.2.4. **If the correlation coefficient for a given element fails to meet this criteria, data for that element can not be reported from this calibration**. The problem must be corrected and the instrument recalibrated with an acceptable correlation coefficient.

10.5.1.2.5. The low level standard in the curve must be at or below the laboratory's routine reporting limit.

10.5.1.2.5.1 If a client requested reporting limit is below the laboratory's routine reporting limit and thus below the low level verification standard, the laboratory will discuss with the client, prior to sample analysis, how to proceed with this requirement.

10.6. **Initial Calibration Verification/Low Level Initial Calibration Verification/Initial Calibration Blank (ICV/LLICV/ICB)**

10.6.1. **ICV**

10.6.1.1. Secondary source, used to verify the initial calibration accuracy.

10.6.1.2. Frequency: Perform with each initial calibration

10.6.1.3. Criteria: ± 10%

10.6.1.4. Action upon failure:

10.6.1.4.1. If the ICV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.6.1.4.2. For all other non-conformances, the samples must be re-analyzed.

10.6.2. **LLICV**

10.6.2.1. Applicable to **6020A** and **6020B** only

10.6.2.2. Same source as calibration

10.6.2.3. Frequency: Perform with each initial calibration.

10.6.2.4. Criteria:

10.6.2.4.1. **6020A**: ± 30%

10.6.2.4.2. **6020B**: ± 20%

10.6.2.5. Action upon failure:

10.6.2.5.1. If the LLICV fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.6.2.5.2. If the LLICV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.6.2.5.3. For all other non-conformances, the samples must be re-analyzed.

10.6.3. **ICB**

10.6.3.1. Frequency: An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.

10.6.3.2. Criteria:

10.6.3.2.1. **200.8/6020/6020A**: ± the RL from zero.

10.6.3.2.2. **6020B**: ± ½ the RL from zero.

10.6.3.3. Action upon failure:

10.6.3.3.1. If the ICB fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.6.3.3.2. If the ICB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.6.3.3.3. For all other non-conformances, the samples must be re-analyzed.

10.6.4. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.

10.6.5. Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.7. **Continuing Calibration Verification/Low Level Continuing Calibration Verification/Continuing Calibration Blank (CCV/LLCCV/CCB)**

10.7.1. Calibration is monitored throughout the analytical run through the analysis of a known mid-level calibration standard.

10.7.2. **CCV**

10.7.2.1. A CCV may be a second source or the same source as the calibration.

10.7.2.2. Frequency: Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run.

10.7.2.3. Criteria: 10% of true value

10.7.2.4. Action upon failure:

10.7.2.4.1. If the CCV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.7.2.4.2. For all other non-conformances, the samples must be re-analyzed.

10.7.3. **LLCCV**
- 10.7.3.1. Applicable to **6020A** only
- 10.7.3.2. Same source as calibration
- 10.7.3.3. Frequency: Perform at a minimum at the end of the run
  - 10.7.3.3.1. Typically analyzed every 10 samples and at the end of the run.
- 10.7.3.4. Criteria: ± 30%
- 10.7.3.5. Action upon failure:
  - 10.7.3.5.1. If the LLCCV fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.
  - 10.7.3.5.2. If the LLCCV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
  - 10.7.3.5.3. For all other non-conformances, the samples must be re-analyzed.

10.7.4. **CCB**
- 10.7.4.1. Frequency: A CCB is analyzed immediately following each CCV.
- 10.7.4.2. Criteria: The CCB result must fall within ± RL from zero.
- 10.7.4.3. Action upon failure:
  - 10.7.4.3.1. If the CCB fails high, but the concentration the associated samples is less then the RL or greater then 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
  - 10.7.4.3.2. If the CCB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
  - 10.7.4.3.3. For all other non-conformances, the samples must be re-analyzed.

10.7.5. If a CCV and/or CCB has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then a second CCV and/or CCB may be analyzed without any adjustments to the instrument. If this CCV and/or CCB meet criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV and/or CCB does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e., initial calibration).

10.8. **Interference Check Standard (ICSA/ICSAB) / Spectral Interference Check (SIC)**
- 10.8.1. Applicable to **6020, 6020A** and **6020B** only
- 10.8.2. For all interference checks, the interfering analyte must be analyzed at its upper linear range.
- 10.8.3. **ICSA**:
  - 10.8.3.1. The ICSA contains only interfering analytes and must include chloride, iron, and molybdenum. Refer to LIMS for the details of the ICSA composition.
  - 10.8.3.2. Frequency: The ICSA must run with each initial calibration or every 12 hours whichever is shorter.
  - 10.8.3.3. Criteria:
    - 10.8.3.3.1. For interfering analytes: ±20%
    - 10.8.3.3.2. For non-interfering analytes: ± 2x RL from zero.
  - 10.8.3.4. Action upon failure:
    - 10.8.3.4.1. For interfering elements:
      - 10.8.3.4.1.1 If the ICSA fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
    - 10.8.3.4.2. For non-interfering elements:
      - 10.8.3.4.2.1 If the ICSA fails high, but the concentration the associated

samples is less then the RL or greater then 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.8.3.4.2.2 If the ICSA fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.8.3.4.3. For all other non-conformances, the samples must be re-analyzed.

10.8.4. **ICSAB**:

10.8.4.1. The ICSAB contains analytes and interferents.

10.8.4.2. Refer to LIMS for the details of ICSAB composition.

10.8.4.3. Custom multi-element ICS solutions may be used.

10.8.4.4. Frequency: The ICSAB must run with each initial calibration or every 12 hours whichever is shorter.

10.8.4.5. Criteria: The ICSAB results must fall within 80% – 120% of the true value.

10.8.4.6. Action upon failure:

10.8.4.6.1. If the ICSAB fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.8.4.6.2. For all other non-conformances, the samples must be re-analyzed.

10.9. **Liner Dynamic Range (LDR)**

10.9.1. Prior to running the instrument, the upper limit of quantitation must be established for each analyte.

10.9.2. This upper limit is tested by running a standard containing high concentrations of the analytes against a calibration curve.

10.9.3. The concentration of the LDR standard can be higher than the high calibration standard.

10.9.4. Frequency:

10.9.4.1. **200.8**: as needed

10.9.4.2. **6020/6020A**: every 6 months

10.9.4.3. **6020B**: daily

10.9.4.4. When requested by client, the LDR is run daily

10.9.5. Criteria: ± 10%

10.9.5.1. If the LDR fails the criteria, the highest calibration standard is used as the upper limit for the linear range.

10.10. **Calibration Sequence**

Tuning Standard
Initial Calibration (3 standards plus a blank)
ICV
ICB
LLICV (for 6020A and 6020B only)
ICSA*
ICSAB*
LDR (when necessary)
CCV
CCB
10 samples (analysis runs)
CCV
CCB
10 samples (repeat every 10 analysis runs)

LLCCV** (for 6020A only)
CCV
CCB
End

* If sequence time is longer than 12 hours, the ICSA and ICSAB standard must be re-analyzed.
**Minimum at end of run. May be analyzed more frequently.

11.0 PROCEDURE

11.1 The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples for Metals.

11.2 The soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils

11.3 Instrument conditions, including rinse times, must be the same for all standards and samples.

11.4 When necessary, dilute samples with 2%HCl / 2% HNO_3. Acid strength in samples must be maintained.

11.5 Internal standards are introduced to the standards and sample digestates by the instrument.

11.6 Load autosampler with standards and digestates in accordance with the sequence given in section 10

11.7 Print sample runlog. The following should be recorded at the top:
- 11.7.4 File name
- 11.7.5 Internal Standard ID
- 11.7.6 Tune Solution ID
- 11.7.7 Dilution Water ID
- 11.7.8 Initials and Date

11.8 Analyze samples.

11.9 A minimum of three exposures for each standard, field sample and QC sample is required. The average of the exposures is reported.

11.10 When analysis is completed, return unused digestate to proper storage area.

11.11 Upload data to LIMS, attaching the raw data and the runlog to the analytical batch.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1. Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2. All measurements must fall within the defined linear range where spectral interference correction factors are valid.
- 12.2.1. Dilute and reanalyze all samples for required analytes that exceed the linear range
  - 12.2.1.1. For 200.8 Drinking Water - any sample greater than 90% of the linear range must be diluted and re-analyzed.
- 12.2.2. Acid strength must be maintained in the dilution of samples.

12.3. The mass ions used for determination of the element of interest is given in Table 1 of this SOP

12.4. Interference equations

12.4.1. Agilent 7500

Mass	Equation
6	(6)*1 – (7)*0.082
111	(111)*1 – (95)*0.00025
115	(115)*1 – (118)*0.014
208	(208)*1 + (206)*1 + (207)*1

12.4.2. Agilent 7700

Mass	Equation
6	(6)*1 – (7)*0.082
111	(111)*1 – (95)*0.00027
115	(115)*1 – (118)*0.014
208	(208)*1 + (206)*1 + (207)*1

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1. The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process in LIMS. The NCM process is described in SOP: ST-QA-0036.

13.2. Method Blank (MB)

13.2.1. Acceptance Criteria:

13.2.1.1. **200.8/6020/6020A**: <RL

13.2.1.2. **6020B**: < ½ RL

13.2.2. Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted in the client notes.

13.2.3. Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.3.1. Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3. Laboratory Control Sample (LCS)

13.3.1. Acceptance Criteria: All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.

13.3.2. Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1. LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2. LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1. Analytes should be within control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.

13.4.2. Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1. MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration

13.4.2.1.1. If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.4.2.1.2. If the excursion is due to physically evident matrix interference, the data is reported with an NCM.

13.4.2.1.3. In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with an NCM.

13.4.2.1.4. When the MS/MSD recoveries and the %RPD are outside criteria, if the samples are non-homogenous, the data may be reported with an NCM. Otherwise, the batch is re-prepped and re-analyzed for the affected analytes.

13.5. Dilution Test (SD)

13.5.1. A dilution test is not required for method 200.8

13.5.2. For methods 6020,6020A and 6020B, a dilution test is analyzed with every prep batch

13.5.3. Criteria:

13.5.3.1. **6020/6020A:** when analyte concentration ≥ 10x RL, ± 10%

13.5.3.2. **6020B:** when analyte concentration ≥ 25x RL, ± 20%

13.5.4. Corrective Action: Dilution Test failure is documented in an NCM and the reported data is flagged. If multiple analytes fail the Dilution Test, the analyst may re-prep and re-analyze the samples.

13.6. Post Digestion Spike (PDS)

13.6.1. A PDS is not required for 200.8.

13.6.2. **6020**

13.6.2.1. Criteria: The acceptance criteria is 75%-125%, UNLESS, other project/program criteria is given.

13.6.2.2. Corrective Action: Sample must be diluted and re-analyzed to compensate for matrix effect, until the PDS is within acceptable limits.

13.6.3. **6020A**

13.6.3.1. Criteria: The acceptance criteria is 80%-120%, with a spike concentration between 10-100 times the MDL, UNLESS, other project/program criteria is given.

13.6.3.2. Corrective Action: A failed PDS is documented with an NCM and noted in the report narrative.

13.6.4. **6020B**

13.6.4.1. Criteria: The acceptance criteria is 75%-125%

13.6.4.2. Corrective Action: A failed PDS is documented with an NCM and noted in the report narrative.

13.7. Sample result evaluation

13.7.1. Dilutions

13.7.1.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.7.2. For samples requiring dilution an NCM is created to document the reason for the dilution.

13.7.3. Insufficient Sample

13.7.3.1. For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative.

13.8. Internal Recovery Standard (IS)

13.8.1. Criteria (for all samples and QC standards)

13.8.1.1. **6020:**

13.8.1.1.1. QC: 80-120%

13.8.1.1.2. Samples: 30-120%

13.8.1.2. **6020A**: 70%-140%

13.8.1.3. **6020B**: 30%-150%

13.8.1.4. **200.8**: 60%-125

13.8.2. Action Upon Failure

13.8.2.1. Samples: If the criteria is not met, the sample should be diluted and re-analyzed until the IS recoveries are within specified limits.

13.8.2.2. QC standards: If the criteria are not met, the analyst will review the data. If the sample internal standard recoveries are within control and the QC standard is within acceptable limits, it is apparent that whatever interference affected the internal standard for the QC standards has not affected the element bracketed by that internal standard based upon the criteria being met. If these specific occurrences are met then an NCM will be generated stating why the data is acceptable. Otherwise, samples linked to the QC standard will be re-analyzed

14.0 METHOD PERFORMANCE

14.1. Method performance data, Reporting Limits, and QC acceptance limits are in LIMS.

14.2. Instrument Detection Limits (IDL's) must be established annually. See Appendix III.

14.3. Demonstration of Capability

14.3.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.4. Training Qualification

14.4.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.4.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4.3. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1. Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2. Waste Streams Produced by the Method

16.2.1. The following waste streams are produced when this method is carried out.

16.2.1.1. Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.1.2. Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid Rad waste for disposal by the

EH&S Coordinator.

17.0 REFERENCES

17.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 6020, 6020A and 6020B

17.2. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma/Mass Spectrometry Method 200.8

17.3. Agilent 7500 Series MassHunter Workstation (G7200A) Operators Manual

17.4. Agilent 7500 Series ICP-MS Hardware Manual

17.5. Agilent 7700 Series ICP-MS Hardware Maintenance Manual

17.6. TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.7. TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.8. Associated SOPs, current revisions:

17.8.1. ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS
17.8.2. ST-IP-0013, Acid Digestion of Aqueous Samples for Metals, SW-846 Method 3005A, 3010A, EPA 200.7 and EPA 200.8
17.8.3. ST-QA-0002, Standard and Reagent Preparation
17.8.4. ST-PM-0002, Sample Receipt and Chain of Custody
17.8.5. ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.8.6. ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.8.7. CA-Q-S-006, Detection and Quantitation Limits
17.8.8. ST-MT-WI-0001, ICPMS Standards
17.8.9. ST-MT-WI-0003, ISOU Standards

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1. Modifications from Method 200.8:

18.1.1. TestAmerica St. Louis digests all aqueous samples. Method 200.8 allows for the digestion step to be omitted under certain conditions.
18.1.2. Method 200.8 requires that data less than 10ug/L be reported to 2 significant figures, and data greater than or equal to 10ug/L be reported to 3 significant figures. TestAmerica St. Louis uses client requested significant figures for reporting data.
18.1.3. Method 200.8 has more stringent blank acceptance criteria than stated in this SOP. Due to software limitations the lab is unable to evaluate and report blanks using the method criteria that “analyte concentration in the blank greater than 2.2 times the MDL, or greater than 10% of the concentration of a sample requires rep-reparation and reanalysis.” Blanks are evaluated to the MDL and flagged if they exceed that level. Re-preparation and re-analysis are required if the method blank is greater than RL and the sample is greater than the RL but less than ten times the RL.
18.1.4. Method 200.8 requires that calibration standards are prepared every two weeks. Instead, TestAmerica St. Louis prepares the ICV standard daily, which is used to show that the calibration standards are still viable. Calibration standards are prepared at a minimum of every 6 months, or more frequently if needed.

18.2. Modifications from Method 6020A:

18.2.1. Method 6020A requires that method blanks are less than project specific DQO’s. This SOP states that the method blank should be less than the RL.
18.2.2. Method 6020A requires the analysis of a Lower Limit Quantitation Check Sample (LLQC) on an as

needed basis, to establish and confirm the lowest quantitation limit. TestAmerica St. Louis fills this requirement with the quarterly running of a MDL verification standard which is taken through the entire sample preparation procedure.

18.2.3. Method 6020A stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed. Internal standards are used to monitor matrix interferences in all samples. Post spikes are done per specific QAPP or program requirements. Post-spikes using analytes other than the internal standards may be used if an analyst encounters a new or unusual matrix.

18.3. Modifications from Method 6020B:

18.3.1. Method 6020B requires that the method blank does not contain target analytes at concentration levels that exceed the acceptance limits defined in Chapter One of SW-846, or in the project-specific DQO's. It then goes on to say that "blanks are generally considered acceptable if the target analyte concentrations are less than ½ the LLOQ or are less than the project-specific requirements". This SOP states that the method blank should be less than ½ RL.

19.0 CHANGES TO PREVIOUS REVISION

19.1. Updated formatting and spelling errors throughout SOP
19.2. Updated section 4.4 referring to the amount of an internal standard being used.
19.3. Added new instrument and gases used in section 6.0.
19.4. Added Lithium to section 7.0 as part of the new reagents and standards used.
19.5. Made reference to new instruments for calibration in section 10.0.
19.6. Add new list of tuning element for both instruments in section 10.5.
19.7. Updated the internal standard intensity throughout section 10.7 and section 10.8
19.8. Added new elements to table 2.
19.9. Rev. 18;
19.9.1. Added LLICV to definitions in section 3.2.
19.9.2. Removed Hydrogen Peroxide from Safety Section (included in prep SOP)
19.9.3. Added tuning solution to section 7.5.5.
19.9.4. Updated cone conditioning solution, make up and frequency of use.
19.9.5. Added clarification to tuning section 10.5.
19.9.6. Added Low Level initial calibration verification standards plus criteria to section 10.0.
19.9.7. Updated tables 1 and 2, added analytes, updated concentrations.
19.9.8. Added method 1311 MSA requirements information to section 9.7.
19.9.9. Spelling and grammatical corrections.
19.10. Rev. 19:
19.10.1. Updated Table III regarding QC Criteria limits.
19.11. Revision 20:
19.11.1. Updated section 1.3 adding reference to the Technetium-99 soil procedure.
19.11.2. Added formulas for determining the Tracer Recovery and the Final tracer Corrected Concentration to section 12.5.
19.11.3. Added instrument software and hardware to section 6.0.
19.11.4. Updated the PDS acceptance criteria in section 9.6.
19.12. Rev. 21:
19.12.1. Removed legacy text regarding MSA from Section 18 as MSA is not required by Method 6020A.
19.13. Rev. 22: (8/27/2013)
19.13.1. Updated section 1.3 to reflect the corrected Tc-99 SOP (ST-RC-0125)
19.13.2. Updated section 6. Hydrogen was removed
19.13.3. Updated section 9.7, replace QuantIMS wording with TALS wording
19.13.4. Updated section 10, used a more consistent format
19.13.5. Updated section 12.4. Removed references to a spreadsheet, added TALS
19.13.6. Updated section 13
19.14. Rev.23: (1/16/2015)
19.14.1. Made formatting and grammatical corrections

19.14.2. Corrected definitions in section 3 (removed IPC and CRI, added LRB)
19.14.3. Removed references to ASTM Method D5673-03
19.14.4. Added equipment and software for ICPMS 9000 to section 6
19.14.5. Removed the ICSA/ICSAB Table (was Table 2) and replaced references to it with instructions to look at LIMS (10.8.3.1 and 10.8.4.2)
19.14.6. Updated tuning criteria for Perkin Elmer ICPMS in sections 10.5.3.1.4 & 10.5.3.1.5
19.14.7. Updated section 10.8.3.5 , replace RL > 10ug/L with RL ≥10ug/L
19.14.8. Clarified criteria in section 10, 13 and 18
19.14.9. Replaced "client requirement sheet" with "client notes" in section 13.2.2
19.14.10. Removed reference to Marginal Exceedance in section 13.3
19.14.11. Corrected IS criteria in section 13.8.1
19.14.12. Updated instrument manuals in section 17
19.14.13. Added affected methods to section 18
19.14.14. Removed section 18.3, was same as section 18.4
19.14.15. Added reference to method 6020
19.14.16. Deleted references in section 9 to batch QC criteria, referenced section 13. Instrument QC criteria is in section 10.
19.14.17. updated formatting, to make the wording more consistent throughout the SOP and easier to read.
19.14.18. section 10: separated out the tuning for Perkin Elmer vs Agilent, for clarity.
19.14.19. Added reference to tc-99 prep via ST-RC-0125
19.15. Rev. 24: (06/22/2015)
19.15.1. Added Appendix 1 MSA instruction
19.16. Rev. 25: (08/11/2016)
19.16.1. Added Appendix II – Retrieve Lost Data from Agilent 7500 or 7700
19.16.2. Added method 6020B and all it's requirements
19.16.3. Updated Table 2
19.16.4. Updated definitions
19.16.5. Updated frequency needed to make standards
19.16.6. Made clarifications throughout
19.16.7. Changed wording from "serial dilution" to "dilution test"
19.16.8. Updated criteria for ICSA
19.16.9. added method modification to section 18: MB for 200.8 and sig figs for 200.8
19.16.10. updated matrix for water MB and LCS
19.17. Rev 26 (6/8/17)
19.17.1. Added corrective action for calibration failure to Section 10.5.1
19.17.2. Updated MSDS to SDS in Section 5
19.18. Rev 27 (2/12/2018) Technical Review – C. Buffington; QA Review – K. Ely
19.18.1. Section 7.2: corrected DI criteria
19.18.2. Section 10.1: Corrected primary tuning solution (to ICSA solution)
19.18.3. Section 10.5.1.2: clarified that 200.8 does not have calibration criteria
19.18.4. Section 12.5: added interference equations
19.18.5. Created Appendix III for sample screening (referenced in section 11)
19.19. Rev 28 (2/1/2019) Technical Review – F.Cruz ; QA Review – K. Ely
19.19.1. Section 1.3: Added prep method 3005A and removed reference to Tc-99
19.19.2. Section 6: Removed software versions
19.19.3. Section 7:
19.19.3.1. Added 2% HCL / 2% HNO_3
19.19.3.2. Added where to find recipe for standards.
19.19.3.3. Added the requirement to list the IS standard and the Tuning standards on the instrument runlog
19.19.3.4. Added 6020B requirements for ICSA/ICSAB (frequency)
19.19.4. Section 9
19.19.4.1. Clarified that limits can be found in Section 13 or LIMS
19.19.4.2. Section 9.2 and 9.3: added Teflon chips as an option for solid matrix.
19.19.5. Section 10

19.19.5.1. Added 30 minutes for warm up
19.19.5.2. Changed frequency of cone conditioning from “daily” to when cones are cleaned or replaced.
19.19.5.3. Section 10.9
19.19.5.3.1. Added SIC
19.19.5.3.2. Added that interfering analytes must be spiked at upper linear range
19.19.5.3.3. Added criteria for interfering analytes.
19.19.5.3.4. Added the a multi analyte standard “may” be used, not “must”
19.19.5.4. Section 10.10: clarified that the LDR can be over the upper calibration range.
19.19.5.5. Section 10.11: clarified that the LLICV is also for 6020B, and that the LLCCV is for 6020A only.
19.19.6. Section 11
19.19.6.1. Added that when necessary, dilute sample, keeping acid strength
19.19.6.2. Added documentation requirements for instrument runlog
19.19.6.3. Added requirement for 3 exposures.
19.19.6.4. Added that after analysis, upload data to LIMS and attach raw data to the analytical batch.
19.19.7. Section 14: added IDL requirement
19.19.8. Section 17: removed reference to ST-QA-0016, added reference to CA-Q-S-006 (Detection and Quantitation Limits)
19.19.9. Appendix IV: Added, instruction for IDL determination.
19.20. Added Eurofins logo and updated copyright information (4/18/2019)
19.21. Rev 29 (11/1/2019) Technical Review – C. Buffington ; QA Review – K. Ely
19.21.1. Removed references to the Perkin Elmer instrumentation
19.21.2. Section 7 – added reference to work instruction for ISO-U standards
19.21.3. Section 11 – added requirement to record dilution water ID at top of runlog
19.21.4. Section 12 – removed references to tracer.
19.21.5. Section 17
19.21.5.1. Removed reference to SOP ST-RC-0125 (Tc-99 prep)
19.21.5.2. Added reference to work instruction ST-MT-WI-0003 (ISOU standards)
19.21.6. Section 18 – added method mod for 200.8, on how often standards are made.
19.21.7. Removed Appendix III (sample screening on Perkin Elmer instruments)
19.21.8. Renamed Appendix IV (IDL’s) to Appendix III

Table 1
ANALYTICAL ISOTOPES

ELEMENT	7700 Tune Step	7500 Mass	7700 Tune Step	7700 Mass	6100 Mass	9000 Mass
Li	3	7	3	7	N/A	N/A
Be	3	9	3	9	N/A	N/A
B	3	11	3	11	N/A	N/A
Na	2	23	2	23	N/A	N/A
Mg	2	24	2	24	N/A	N/A
Al	2	27	2	27	N/A	N/A
Si	3	28	3	28	N/A	N/A
P	2	31	2	31	N/A	N/A
S	2	34	2	34	N/A	N/A
K	2	39	2	39	N/A	N/A
Ca	3	44	3	44	N/A	N/A
Ti	3	47	3	47	N/A	N/A
V	2	51	2	51	N/A	N/A
Cr	2	52	2	52	N/A	N/A
Mn	2	55	2	55	N/A	N/A
Fe	2	57	2	57	N/A	N/A
Co	2	59	2	59	N/A	N/A
Ni	2	60	2	60	N/A	N/A
Cu	2	63	2	63	N/A	N/A
Zn	2	66	2	66	N/A	N/A
As	2	75	2	75	N/A	N/A
Se	2	78	2	78	N/A	N/A
Sr	3	88	3	88	N/A	N/A
Y	2	89	2	89	N/A	N/A
Zr	2	90	2	90	N/A	N/A
Nb	2	93	2	93	N/A	N/A
Mo	3	95	3	95	N/A	N/A
Ru	2	101	2	101	N/A	N/A
Rh	2	103	2	103	N/A	N/A
Pd	2	105	2	105	N/A	N/A
Ag	3	107	3	107	N/A	N/A
Cd	3	111	3	111	N/A	N/A
Sn	3	118	3	118	N/A	N/A
Sb	3	121	3	121	N/A	N/A
Te	2	125	2	125	N/A	N/A
Cs	2	133	2	133	N/A	N/A
Ba	3	137	3	137	N/A	N/A
La	2	139	2	139	N/A	N/A
Ce	2	140	2	140	N/A	N/A
Pr	2	141	2	141	N/A	N/A
Nd	2	146	2	146	N/A	N/A
Sm	3	147	3	147	N/A	N/A
Hf	2	178	2	178	N/A	N/A
Ta	2	181	2	181	N/A	N/A
W	2	182	2	182	N/A	N/A
Re	3	185	3	185	N/A	N/A
Pt	2	195	2	195	N/A	N/A
Au	3	197	3	197	N/A	N/A
Tl	3	205	3	205	N/A	N/A
Pb	3	208	3	208	N/A	N/A
Bi	2	209	2	209	N/A	N/A
Th	3	232	3	232	N/A	N/A
Tc	3	99	N/A	N/A	99	99
U	N/A	N/A	3	236	236	236
U	N/A	N/A	3	235	235	235
U	N/A	N/A	3	234	234	234
U	N/A	N/A	3	233	233	233
U	3	238	3	238	238	238

Tune Step 2: Helium
Tune Step 3: No Gas (argon only)

Table 2
QC Criteria

Methods	6020	6020A	6020B	200.8
Corr Coeff.	>0.998	>0.998	>0.995	N/A
Tuning Res	<0.9amu 10% peak height	<0.9amu 10% peak height	<0.9amu 10% peak height	≈ 0.75amu 5% peak height
Int Std	QC: 80-120% Samples: 30-120%	70-140%	30-150%	60-125%
ICV	90-110%	90-110%	90-110%	90-110%
ICB	±RL	±RL	± ½ RL	±RL
LLICV	N/A	70-130%	80-120%	N/A
LLCCV	N/A	70-130%	N/A	N/A
CCV	90-110%	90-110%	90-110%	90-110%
CCB	±RL	±RL	±RL	±RL
MB	±RL	±RL	± ½ RL	±RL
LCS	80-120%	80-120%	80-120%	85-115%
MS	75-125%	75-125%	75-125%	70-130%
PDS	75-125%	80-120%	75-125%	N/A
SD	± 10%	± 10%	± 20%	N/A

Table 3

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS
BACKGROUND MOLECULAR IONS

Molecular Ion	Mass	Element Interferences*
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H_+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	
$^{40}ArH^+$	41	
CO_2^+	44	
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	
$ArOH^+$	57	
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se

* Method elements or internal standards affected by the molecular ions.

Table 4

MATRIX MOLECULAR IONS* No gas Mode Only

CHLORIDE		
Molecular Ion	Mass	Element Interference
$^{35}Cl0^+$	51	V
$^{35}Cl0H^+$	52	Cr
$^{37}Cl0^+$	53	Cr
$^{37}Cl0H^+$	54	Cr
$Ar^{35}Cl^+$	75	As
$Ar^{37}Cl^+$	77	Se

SULFATE		
Molecular Ion	Mass	Element Interference
$^{32}SO^+$	48	
$^{32}SOH^+$	49	
$^{34}SO^+$	50	V, Cr
$^{34}SOH^+$	51	V
SO_2^+, S_2^+	64	Zn
$Ar^{32}S^+$	72	
$Ar^{34}S^+$	74	

PHOSPHATE		
Molecular Ion	Mass	Element Interference
PO^+	47	
POH^+	48	
PO_2^+	63	Cu
ArP^+	71	

GROUP I, II METALS		
Molecular Ion	Mass	Element Interference
$ArNa^+$	63	Cu
ArK^+	79	
$ArCa^+$	80	

MATRIX OXIDES*		
Molecular Ion	Mass	Element Interference
TiO	62-66	Ni, Cu, Zn
ZrO	106-112	Ag, Cd
MoO	108-116	Cd

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

APPENDIX 1 – METHOD OF STANDARD ADDITION (MSA)

MSA is required if

1- Matrix spike recovery <50%, AND
2- Measured concentration is within 20% of the regulatory level

Regulatory Limits:

Arsenic	5mg/L
Barium	100mg/L
Cadmium	1 mg/L
Chromium	5 mg/L
Lead	5 mg/L
Mercury	0.2 mg/L
Selenium	1 mg/L
Silver	5 mg/L

How to run an MSA

1- Take 4 identical aliquots of test solution
2- Add increasing concentration of standard to 3 aliquots and add blank solution to 4th aliquot- all aliquots should be at same final volume
3- Perform analysis
4- Enter data into spreadsheet ORG-0034_TCLP_MSA
    a. Uses unweighted least-squares linear regression curve fit
    b. Calculates absolute value of x-intercept

Notes:

1- MSA spikes must come from the same analytical batch, analyzed on the same day.

The MSA curve must include the unspiked sample and 3 samples spiked with increasing concentrations of analytes

APPENDIX II- Retrieve Lost Data from Agilent 7500 or 7700

MASS HUNTER

File-> New Batch Folder (create, give new name)
File-> Import All Samples from Batch

DA Method Editor

Import DA Method and Standard Data (METALSTA – Met_2014)
Return to Batch-at-a glance
Update? YES

Process Batch

APPENDIX III – IDL Determination

1.0 Instrument Detection Limit(IDL): The instrument detection limit is defined as the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument.

2.0 Instrument Detection limits are established for new instrumentation, after major maintenance, as designated by a project or at a minimum, annually.

3.0 IDL Procedure

3.1 IDL Requirements

3.1.1 Instrument detection limits (IDLs) are typically used in metals analysis to evaluate the instrument noise level and response changes over time for analytes of interest. IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project. IDLs are performed for methods 6010D and 6020B at a minimum annually

3.2 IDL Analysis

3.2.1 Instrumentation must be configured with the identical settings used for routine sample analysis for which the IDL is applicable.

3.2.2 Instrumentation must be calibrated according to method specifications and SOP requirements.

3.2.3 Analyze 10 instrument blanks (e.g. calibration blank solution). IDLs can be determined in a single analytical run.

3.2.4 Each measurement should be performed as though it were a separate calibration standard (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples)

3.3 IDL Calculation

3.3.1 IDLs in µg/L can be estimated as the mean of the blank results plus three times the standard deviation of 10 replicate analyses of the calibration blank solution. (Use zero for the mean if the mean is negative).

3.3.2 Determine the mean and the standard deviation of the ten replicates

3.3.3 Instrument Detection Limits are calculated as follows:

$$IDL = mean + (3* Std\ Dev)$$

3.4 Evaluate the IDL

3.4.1 The calculated IDL must be evaluated to determine if a valid number has been obtained.

3.4.2 The IDL should be less than the MDL.

3.4.2.1 If the IDL is greater than the MDL, evaluate instrument performance, performing maintenance (if applicable), and reanalyze the IDL study.

3.4.2.2 If IDL result is still greater than MDL, instrument signal noise is interfering with detection, elevate the MDL

Title: DETERMINATION OF GROSS ALPHA/BETA ACTIVITY

Approvals (Signature/Date):

Chelsea Mazariegos 11/05/2019
Chelsea Mazariegos — Date
Radiochemistry Prep Manager

Michael Ridenhower 11/6/19
Michael Ridenhower — Date
Health & Safety Manager / Coordinator

Kristen Ely 11/6/19
Kristen Ely — Date
Quality Assurance Manager

Sarah Bernsen 11/5/19
Sarah Bernsen — Date
Radiochemistry Operations Manager

This SOP was previously identified as SOP No. ST-RC-0020 Rev. 21

Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1 This procedure applies to the preparation and analysis of samples for gross alpha and/or beta radioactivity in air filters, water, soil/sediment, oil and vegetation samples.

1.2 This SOP is based on EPA Method 900.0, SW-846 Method 9310 and DOE RP-710.

1.3 For water samples containing high concentrations of dissolved solids (> 500 ppm), see SOP ST-RC-0021 for analysis of gross alpha radioactivity.

1.4 The reporting limits, method detectable activities and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 An aliquot of aqueous sample is evaporated to dryness in a glass beaker after the addition of concentrated nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tared counting planchet.

2.2 For the activity of dissolved matter, an aliquot of aqueous sample is filtered through a 0.45-µm membrane filter. The filtrate is evaporated to dryness in a glass beaker after the addition of concentrated nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tarred counting planchet.

2.3 For the activity of suspended matter, an aliquot of aqueous sample is filtered through a 0.45-µm membrane filter. The filter is transferred to a counting planchet.

2.4 Air filter samples are counted for gross alpha and/or beta activity without further processing if the filter is less than 2 inches diameter. If the filter is greater than 2-inch diameter, the sample is digested per ST-RC-0004, “Preparation of Soil, Sludge, Filter, Biota, Oil and Grease Samples for Actinide Analysis” and then an aliquot prepared like a liquid.

2.5 Solid samples can be analyzed for gross alpha and/or beta activity as a dry powder. If Method RP710 (for total dissolution) is required, an acid leach is performed per ST-RC-0004, “Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis”. The digestate is then treated like a liquid**.**
NOTE: Total Sample Dissolution can also be done using Hydrofluoric acid, Hydrochloric acid and Nitric acid as in section 11.8.

2.6 Oil samples are ashed in a muffle furnace, then dissolved in nitric acid and transferred to a glass beaker where they are converted to nitrate salts using concentrated nitric acid. The sample is then transferred to a planchet using 4 M nitric acid.

2.7 The sample residue is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 In this method for gross alpha and gross beta measurement, the radioactivity of the sample is not separated from the solids of the sample. The solid concentration may adversely affect sensitivity of the method.

4.2 For a 2-inch diameter counting planchet (20 cm^2), an aliquot containing 100 mg of dissolved solids would be the maximum aliquot size for that sample which should be evaporated and counted for gross alpha or gross beta activity.

4.3 Radionuclides that are volatile under the sample preparation conditions of this method can not be measured. Other radionuclides may also be lost during the sample evaporation and drying (such as tritium and some chemical forms of radioiodine). Some radionuclides, such as cesium, polonium, and technetium, may be lost when samples are heated to dull red color, so flaming of planchets is not performed by TestAmerica St. Louis. Such losses are limitations of the test method.

4.4 Moisture absorbed by the sample residue increases self absorption and, if uncorrected, leads to low-biased results. Hygroscopic sample matrices may not remain at a constant weight after being dried and exposed to the atmosphere before and during counting. Those types of water samples are sometimes heated to a dull red color for a few minutes to convert the salts to oxides. This practice is not recommended, as the calibration performed is for the nitrate form (not for the oxide) and a bias to the results will occur. And, as stated in 4.3, such heating can also cause certain isotopes to be lost. For this reason, flaming of planchets is not performed by TestAmerica St. Louis. It is suggested in this instance the sample be reheated on the hot plate to attempt to drive off the added waters of hydration. If the sample afterward still appears to be hygroscopic, it is recommended the sample be reweighed after reaching constant weight (to account for the added mass in the attenuation curve) and then counted. If the sample does not appear to reach constant weight in a reasonable period of time, this should be noted in a NCM as a limitation of the method.

4.5 Heterogeneity of the sample residue in the counting planchet interferes with the accuracy and precision of the method.

4.6 Gross Alpha and Gross Beta activity does not identify the radionuclide that is present. Instead, the activity is referenced as equivalent to Th-230 for Gross Alpha and Sr-90/Y-90 for Gross Beta.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method

can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-(TWA) 4 ppm-(STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-(Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid	Poison Corrosive Dehydrator	3 ppm-(TWA)	Severely corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical Balance (4 - or 5 - place).

6.2 Beakers: Glass and Teflon, various sizes. Please consult SOP: ST-RC-5006 “Decontamination of Laboratory Glassware, Labware, and Equiptment.”

6.3 Counting planchets, stainless steel, flat and ridged, 5.0 cm (2.0"), cleaned per ST-RC-0002, "Preparation of Stainless Steel Planchets for Radiochemistry Analyses."

6.4 Desiccator with desiccant, Dri-Rite or equivalent.

6.5 Drying oven with thermostat set at 105° C ± 5 °C.

6.6 Filter paper: ashless, Whatman #41 or ashless paper pulp, and 0.45-µm membrane.

6.7 Hot plate

6.8 Pipettes

6.9 Muffle oven

6.10 Mod block

6.11 Tongs or forceps

6.12 Double sided tape or Self-adhesive dots

6.13 Spatula

6.14 Aluminum weighing pans

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Reagents are prepared from reagent grade chemicals, unless otherwise specified below, and reagent water.

7.3 Deionized Water

7.4 Nitric acid, concentrated (16M HNO_3)

7.4.1 4 M Nitric acid (4M HNO_3) – Non-critical reagent: Add approximately 250 mL of 16M HNO_3 to approximately 750 mL of DI water. And mix.

7.5 Hydrofluoric acid, concentrated (2 M HF)

7.6 Hydrochloric acid, concentrated (12M HCl)

7.7 Salt Solution: $NaHCO_3$ – 22 g, KCL – 0.80 g, $MgCl_2 \cdot 6H_2O$ – 22 g, Na_2SO_4 – 34.2 g add to 500 mL of DI water. Stir on stir plate until dissolved. Bring final volume up to 1 L with DI.

7.8 Salt, NaCl, granular.

7.9 Thorium-230 for LCS and matrix spikes, calibrated - NIST traceable, diluted to approximately 20 dpm/ml.

7.10 Strontium-90 for LCS and matrix spikes, calibrated - NIST traceable, in equilibrium with Yttrium 90, diluted to approximately 20 dpm/ml.

7.11 Sodium Bicarbonate, $NaHCO_3$ powder.

7.12 Potassium Chloride, KCl

7.13 Sodium Sulfate, $NaSO_4$ crystals

7.14 Magnesium Chloride Hexahydrate, $MgCl_2 \cdot 6H_2O$, crystals

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

8.3.1 The pH of aqueous samples is checked upon receipt by the Sample Control Department. The pH does not require re-checking prior to analysis.

8.3.2 Aqueous samples acidified upon receipt (designated by label on the bottle) do require a check of the pH prior to analysis.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (DU). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water. Prepare a method blank of DI water equivalent to the target volume of 200 mL.

9.2.4 For Soil analyses, the method blank is comprised of salt.

9.2.5 For Oil analyses, the method blank is comprised of shredded filter paper in a crucible.

9.2.6 For non-digested filters, a prepared method blank is provided by the count room.

9.2.7 For leached analyses, the method blank is comprised of the leaching acid.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.3.2 A LCS must be prepared with every sample batch.
9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Strontium 90 for beta and Thorium 230 for alpha. Add 0.7 mL of salt solution for mass.
9.3.4 For Soil analyses, the LCS is comprised of a known solid reference material :National Bureau of Standards, SRM 4353, Rocky Flats Soil #1.
9.3.5 For Oil analyses, the LCS is comprised of shredded filter paper fortified with Strontium 90 for beta and Thorium 230 for alpha.
9.3.6 For non-digested filters, the LCS is provided by the count room.

9.4 **Matrix Spike (MS)/Matrix Spike Duplicate (MSD)**
9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate (DU)**
9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of a LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**
9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance calibration must be checked daily when used. Refer to SOP ST-QA-0005, “Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes Procedure.

10.2 For analytical instrumentation calibration, see SOP: ST-RD-0403, “Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System”.

11.0 PROCEDURE

11.1 If the activity of dissolved matter in an aliquot of aqueous sample is to be determined.
11.1.1 Filter the desired aliquot through a 0.45-µm membrane filter and proceed with aqueous sample preparation.

11.2 If the activity of suspended matter of an aliquot of aqueous sample is to be determined.
11.2.1 Filter the desired aliquot through a 0.45-µm membrane filter, and proceed with filter sample preparation.

11.3 Aqueous Sample - Total Solid Screen
11.3.1 Record sample preparation data in Gross Alpha/Beta (GAB) Solid Screen Excel program (RAD-0052). Weigh the empty beaker and record weight (under the tare weight header)

11.3.2 Shake the sample container thoroughly.
11.3.2.1 If alpha and beta are to be determined simultaneously from a single aliquot, the lowest net residue weight limit applies.
11.3.3 Measure a 20 mL aliquot into a pre-weighed beaker.
11.3.4 Add 10 mL of concentrated Nitric acid.
11.3.5 Evaporate to near dryness using a hot plate, do not allow the sample to splatter.
11.3.6 Remove from heat and allow to cool to room temperature.
11.3.7 Add 10 mL concentrated Nitric acid.
11.3.8 Evaporate to near dryness using a hot plate. Do not allow the sample to splatter.
11.3.9 Remove from heat and allow to cool in desicator for a minimum of 30 minutes.
11.3.10 Reweigh the beaker in GAB Solid Screen Excel (RAD-0052) program and record weight (under the gross weight header)
11.3.10.1 By estimating the solid content of the sample, the program will provide the target aliquot .
11.3.10.2 If GAB Solid Screen Excel program is not available use the formula found in 12.2.
11.3.10.3 From the net residue weight and sample volume used, determine the sample volume required to meet the target residue weight using the formula given in step 12.2, with a target weight of 80 mg alpha/beta dried residue on the planchet **(sample weights should not exceed 100 mg, if sample weights exceed 100 mg an aliquot of the dried residue should be taken after redissolving in 4 M nitric acid. Dilutions are noted on the worksheet. If it is not practical to redissolve the residue the sample should be redone using less volume. If it is not practical to redissolve or restart the sample, check with the count room supervisor or designee to verify that the sample weight fits on the current alpha curve before counting.)**. If only Gross Beta is being performed, the target weight is to 160 mg. Compare the calculated volume to meet the weight limitation with the volume required to ensure that the MDA is below the Reporting Limit. The volume for analysis is the smaller of the two volumes.

11.4 Aqueous Sample Gross Alpha/Beta
11.4.1 Initiate sample preparation worksheet.
11.4.2 Shake the sample container thoroughly.
11.4.3 Measure a volume of sample, previously determined in section 11.3, into an appropriately sized beaker. Record volume of sample used.
11.4.3.1 If it is determined (in step 11.3) that only a small volume of sample is required, additional volume may be added in small aliquots directly to the beaker used to determine the volume needed to achieve the target sample weight.
11.4.4 Prepare a method blank, LCS and MS.
11.4.5 Add 10 mL of concentrated Nitric acid to all samples and QC.
11.4.6 Evaporate to near dryness using a hot plate. Do not allow the sample to splatter.
11.4.7 Remove from heat and allow to cool to room temperature.
11.4.8 Add 10 mL concentrated Nitric acid.
11.4.9 Evaporate to near dryness using a hot plate. Do not allow the sample to splatter.
NOTE: Some samples with difficult matrices may require steps 11.4.7 through 11.4.9 to be repeated until the sample residue does not change in appearance.
11.4.10 Remove from heat and allow to cool to room temperature.
11.4.11 Add 10 mL of 4 M nitric acid to wash down the sides of the beaker.
11.4.12 Heat on hot plate to dissolve sample residue and reduce volume to approximately 5-7 mL.
11.4.13 Transfer the sample to a ridged stainless steel planchet.
11.4.14 Wash down the beaker with small portions of 4M HNO_3 and add to the planchet.
11.4.15 Evaporate planchets to dryness on a hot plate. Do not allow the sample to splatter.

11.4.16 Remove sample planchets from hot plate.
11.4.17 Cool planchets in a desiccator for a minimum of 30 minutes.
11.4.18 If sample appears hygroscopic, the sample may be re-heated on the hot plate in between cooling in a desiccator until a constant weight is found.
11.4.19 Weigh the cooled planchets and record final weight(s).
11.4.19.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.4.19.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.4.19.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.4.20 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.5 Oil Sample
11.5.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.
11.5.2 Fill a 50 mL beaker ¼ full with confetti made from Whatman No. 41 filter paper or ashless paper pulp.
11.5.3 Place beaker on analytical balance, then record the weight in appropriate sample worksheet.
11.5.4 Weigh to the nearest 0.0001 g, approximately 0.1-1gram of the oil sample onto the shredded filter paper. Record the sample weight.
11.5.5 Cover with a crucible lid.
11.5.6 If the sample is a mixture of oil and water or is a sample spiked with an aqueous solution, evaporate the water on a hot plate before muffling. Do not allow residue to "bake" on hot plate. A programmable muffle program may also be used to dry the water before ramping the temperature.
11.5.7 Ramp oven to approximately 600° C and hold there for four hours.
11.5.8 Turn off the muffle oven, crack open the door, and allow the sample to cool to room temperature.
11.5.9 Add approximately 7 mL of 4M HNO_3 to the residue in the beaker.
11.5.10 Transfer the sample to a glass beaker with 4M HNO_3.
11.5.11 Wash down the beaker and lid with small portions of 4M HNO_3 and add to beaker.
11.5.12 Evaporate to near dryness on hot plate. Do not allow sample to splatter. Remove from heat and allow to cool to room temperature.
11.5.13 Add 10 mL of concentrated nitric acid.
11.5.14 Evaporate to near dryness on a hot plate. Do not allow sample to splatter.
11.5.15 Remove from heat and allow to cool to room temperature.
11.5.16 Add 10 mL of 4M nitric acid. Heat to dissolve and then to reduce volume to approximately 5-7 mL.
11.5.17 Transfer sample to a ridged stainless steel planchet.
11.5.18 Wash down the beaker with small portions of 4MHNO_3 and add to the planchet
11.5.19 Evaporate to dryness on a hot plate, do not allow the sample to splatter.
11.5.20 Remove sample from hot plate.
11.5.21 If sample appears hygroscopic, dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours. If not hygroscopic proceed to step 11.5.22.
11.5.22 Cool planchets in a desiccator for a minimum of 30 minutes.
11.5.23 Weigh the cooled planchets and record final weight.

11.5.23.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.5.23.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.5.23.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).

11.5.24 Store dry sample planchets in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.6 Filter Samples

11.6.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.

11.6.2 If the filter is less than 2" diameter, secure the air filter in a stainless steel planchet with double-sided cellophane tape such that no portion of filter extends above the lip of the planchet. Then proceed to step 11.6.20.

11.6.3 If the filter is 2" diameter, the sample can be placed directly in the dectector.

11.6.4 If the filter is greater than 2" diameter, digest or leach the sample per ST-RC-0004 for shared filters. Prepare a method blank and LCS from blank filters, spiked as per 9.3.3, which are digested in the same manner as the samples.

11.6.5 Shake the digested sample thoroughly. Measure a volume of sample into an appropriately sized teflon beaker. Record volume of sample used.

11.6.6 Add 10 mL of concentrated nitric acid.

11.6.7 Evaporate to near dryness on a warm hot plate. Do not allow the sample to splatter.

11.6.8 Remove from heat and allow to cool to room temperature.

11.6.9 Add 10 mL of concentrated nitric acid.

11.6.10 Evaporate to near dryness on a warm hot plate, do not allow the sample to splatter.

11.6.11 Remove from heat and allow to cool to room temperature.

11.6.12 Add 10 mL of 4M nitric acid.

11.6.13 Heat to dissolve and then to reduce volume to approximately 5-7 mL

11.6.14 Transfer the sample to a pre-weighed, stainless steel planchet.

11.6.15 Wash down the beaker with small portions of 4M HNO_3 and add to the planchet.

11.6.16 Evaporate to dryness on a warm hot plate. Do not allow the sample to splatter.

11.6.17 If sample appears hygroscopic dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours. If not hygroscopic proceed to step 11.6.18.

11.6.18 Cool planchets in a desiccator for a minimum of 30 minutes.

11.6.19 Weigh the cooled planchets and record final weight.

11.6.19.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.6.19.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.6.19.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).

11.6.20 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.7 Solid and/or Soil Samples by Dry, Grind Sprinkle

11.7.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.

11.7.2 If the sample has already been prepared per ST-RC-0003, "Drying and Grinding of Soil and Solid Samples," proceed to step 11.7.8 for direct sample mounting.
11.7.3 Use table salt for the blank and a soil standard reference material, e.g. NIST Traceable Rocky Flats Soil, for the LCS. Prepare in the same fashion as the samples.
11.7.4 Remove an aliquot (typically 1 - 5 g.) with a spatula and place into a clean, labeled aluminum weighing pan.
11.7.5 Place sample on a hot plate or in a drying oven at approximately 105° C and evaporate any moisture.
11.7.6 When dry, remove from hot plate or oven and allow the sample to cool.
11.7.7 If necessary, using a metal spatula, reduce the solid sample to a fine particle size.
11.7.8 Use double sided tape to secure the self-adhesive dots (adhesive side up) to a flat stainless steel planchet. Self adhesive label dots are used to hold finely divided solid material uniformly for gross alpha and/or beta analysis. Weigh and record the prepared planchet.
11.7.9 Distribute the sample evenly in the stainless steel planchet.
11.7.10 Record final weight. The target mass is 40-100 mg.
11.7.10.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.7.10.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.7.10.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.7.11 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.8 Solid and/or Soil Samples by total dissolution.
11.8.1 Initiate sample preparation sheet.
11.8.2 Weigh 1.0g sample into a 50 mL beaker and record weight.
11.8.3 Place in oven at 600° and allow to muffle for four hours. Allow to cool.
11.8.4 Transfer to digestion tube using 4M HNO_3.
11.8.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
11.8.6 Digest in mod block at > 110°C for approximately four hours or until dry.
11.8.7 Carefully add 5 mL concnetrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
11.8.8 Digest in mod block at > 110°C for approximately four hours or until dry.
11.8.9 Add 10 mL HNO_3 and digest in mod block at >110°C for approximately 4 hours or until dry.
11.8.10 Reflux samples with 10 mL 4M HNO_3 for 20 minutes using a watchglass over digestion vessel.
11.8.11 Bring up to 20 mL with 4M HNO_3 in the digestion vessel.
11.8.12 Transfer 1 mL of sample to a tared planchet and cook to dryness.
11.8.13 Cool in descicator for 30 minutes.
11.8.14 Reweigh the planchet to determine the mass of 1 mL.
11.8.15 Determine the total amount of sample needed to reach the target mass of 100 mg on the planchet.
11.8.16 Transfer amount of sample to a 250 mL beaker.
11.8.17 Prepare blank, MS and LCS.
11.8.18 Add 10 mL of HNO_3 and Evaporate to near dryness. Allow to cool.
11.8.19 Add 10 mL of 4M nitric acid.

11.8.20 Heat on hot plate to dissolve sample residue and then to reduce volume to approximately 5-7 mL.
11.8.21 Transfer the sample to a ridged stainless steel planchet.
11.8.22 Wash down the beaker with small portions of 4M HNO_3 and add to the planchet.
11.8.23 Evaporate to dryness on a warm hot plate. Do not allow liquid to splatter.
11.8.24 Remove sample from hot plate.
11.8.24.1 If sample appears hygroscopic, dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours.
11.8.25 Weigh the cooled planchets and record final weight.
11.8.25.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.8.25.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.8.25.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.8.26 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.9 Reprocessing planchets which are over the weight limit.
11.9.1 Rinse residue from planchet with 4M HNO_3 into a beaker. Add 4M HNO_3 to planchet and heat if necessary to complete the transfer.
11.9.2 Redissolve the residue into 4M HNO_3. Dilute the sample to a known volume.
11.9.3 Remove an aliquot which will keep the residue weight under the limit (100 mg) and transfer to the pre-weighed planchet. Record information on sample worksheet.
11.9.4 Evaporate to dryness on a warm hot plate so that the sample does not boil.
11.9.5 Remove sample from hot plate. Allow to cool.
11.9.6 Weigh the cooled planchet and record final weight.
11.9.7 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analytical SOP.
12.2 To calculate the aqueous sample volume required (ml), use the following equation:

$$volume\ required\ (mL) = \frac{target\ net\ residue\ weight\ (mg) * initial\ aliquot\ volume\ (mL)}{initial\ aliquot\ net\ residue\ weight\ (mg)}$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analytical SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS

14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification
- 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method
- 16.2.1 The following waste streams are produced when this method is carried out.
  - 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

17.0 REFERENCES

17.1 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," Method 900.0, August, 1980.

17.2 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Method 9310, Rev. 0, September, 1986.

17.3 DOE Method RP-710, "Laboratory Method for Gross Alpha and Beta Activity Determination, 1997

17.4 TestAmerica St. Louis Laboratory Quality Assurance Manual (ST-QAM)

17.5 Corporate Environmental Health and Safety Manual (CW-E-M-001) and Facility addendum.

17.6 Associated SOPs

17.6.1 ST-PM-0002, Sample Receipt and Chain of Custody
17.6.2 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Analyses.
17.6.3 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
17.6.4 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil and Grease Samples for Radiochemical Analysis
17.6.5 ST-RC-0021, Gross Alpha Radiation in Water Using Coprecipitation
17.6.6 ST-RD-0403, Low Background Gas Flow Proportional Counting (GFPC) System
17.6.7 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.6.8 ST-QA-0002, Standards and Reagent Preparation
17.6.9 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.6.10 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated section 9.2.7: added leached analyses use of a method blank that is comprised of leaching acid.
19.2 Replaced piptte with pre-weighted beaker to measure sample aliquot in section 11.3.3.

19.3 Rev 14:
19.3.1 Updated the total dissolution procedure for solid/soil samples in Section 11.8.

19.4 Revision 15:
19.4.1 Added required pH checking for all aqueous samples prior to analysis in section 8.3.
19.5 Revision 16:
19.5.1 Added reference to DOE Method RP-710 to Sections 1 and 17

19.6 Revision 17:
19.6.1 Updated section 15.
19.6.2 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.
19.6.3 Removed references to ‘Clouseau” and “Quantims”, replaced with LIMS.
19.6.4 Updated method requirements for Air Filter samples in section 2.0.
19.6.5 Updated supplies in section 6.0.
19.6.6 Updated reagents and standards in section 7.0.
19.6.7 Replaced the use of a porcelain crucible with a beaker throughout section 11.0.
19.7 Rev.18: (02/14/2014)
19.7.1 Section 8, removed sample hold time
19.7.2 Section 11.3.1, added GAB solid screen forrn Rad-0052
19.7.3 Section 11.1.10, added GAB solid screen form Rad-0052
19.7.4 Grammatical errors fixed throughout
19.8 Rev. 19: (04/24/2015)
19.8.1 Procedure updated in section 11.0
19.9 Annual Review, no changes (05/18/2016)
19.10 Revision 20: (05/12/2017)
19.10.1 Updated section 5: fixed acronym from MSDS to SDS
19.10.2 Updated section 7: clarified how to make reagents
19.10.3 Updated section 9: changed acronym for sample duplicate, from SD to DU, to match LIMS

19.10.4 Updated section 11: fixed acid concentrations

19.11 Revision 21: (01/29/2018)

19.11.1 Technical Review S. Bernsen/QA Review M. Ward

19.11.2 Updated section 11.9- changed 4N to 4M nitric acid.

19.11.3 Grammatical errors fixed through out.

19.12 Annual Review, no changes (01/16/2019)

19.12.1 Technical review S. Bernsen/QA Review M Ward

19.13 Added Eurofins logo and updated copyright information (4/16/2019)

19.14 Revision 22 (11/12/2019)

19.14.1 Technical Review – C. Mazariegos; QA Review – K. Ely

19.14.2 Section 4 – Noted that TA St. Louis does not flame planchets. Also added information as to why this is not done.

19.14.3 Section 9

19.14.3.1 Removed duplicated information about NCM's.

19.14.3.2 Removed information about QC codes (from old LIMS).

19.14.4 Section 11.4 – Corrected laboratory procedure for samples that appear hygroscopic.

19.14.5 Section 17 – Updated SOP names.

Title: PREPARATION OF SAMPLES FOR GAMMA SPECTROSCOPY [EPA 901.1 and DOE GA-01-R]

Approvals (Signature/Date):

Chelsea Mazariegos 11/05/2019
Chelsea Mazariegos Date
Radiochemistry Prep Manager

Mike Ridenhower 11/6/19
Mike Ridenhower Date
Health & Safety Manager / Coordinator

Kristen Ely 11/6/19
Kristen Ely Date
Quality Assurance Manager

Sarah Bernsen 11/5/19
Sarah Bernsen Date
Radiochemistry Operations Manager

This SOP was previously identified as SOP No. ST-RC-0025 Rev. 18

Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1 The purpose of this SOP is to provide detailed instructions for the preparation of samples which require gamma spectroscopy analysis.

1.2 This SOP describes methods for the preparation of samples of liquid, soil, vegetation, air filter, and core matrices prior to gamma spectroscopy analysis.

1.3 This SOP is based on EPA Method 901.1 and DOE Method GA-01-R.

1.4 The laboratory target analytes supported by this method, the reporting limits, and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Samples are transferred to a standard geometry container for counting on the gamma detectors. High purity germanium (HPGe) gamma detectors are used to detect isotopes with gamma ray energies between 40 and 2000 KeV. Activity concentration is determined using commercially available gamma spectral analysis software. A sample matrix which can be mounted in one of the standard geometries may be analyzed for any of the isotopes included in the radionuclide reference library. Detection limits may be affected by the sample size. Gamma photon energies not identified in the reference library may be identified and evaluated manually.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Replicate Analyses – Two or more analysis of the same sample whose independent measurements are used to determine the precision of the equipment's analytical procedure.

4.0 INTERFERENCES

4.1 Gamma energy emissions identified with scientifically measured probability by some radionuclides are documented by multiple sources. There are some discrepancies between reference sources and attempts are made to evaluate the reference data used in spectral analysis. Gamma emissions at discreet energy and probability are used to identify and quantify specific radionuclides in the sample. Gamma emissions which are completely absorbed by an HPGe detector form photo peaks which are used for identification and quantification of gamma emitting radionuclides. When two or more nuclides emit similar gamma energy the photo peaks cannot be resolved without using complex algorithms. These photo peaks in close proximity can interfere with the identification or quantification of a radionuclide. Knowing this, the nuclide reference library, computer software and analyst training are used to minimize the possibility of interference and misidentification. It is not possible to eliminate all interferences and misidentification.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all

samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 **Wear Kevlar or MAPA Blue-Grip gloves when using knives or sharp articles.**

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. ***NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.*** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Nitric acid can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1- Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance, top loader

6.2 Blender

6.3 Food chopper/grinder

6.4 Knives appropriate for food preparation

6.5 Graduated cylinder

6.6 Filter disk, 47 millimeter diameter

6.7 Plastic Tape

6.8 Marinelli beakers of various sizes (500 mL and 1L)

6.9 Petri dishes, 2-inch diameter

6.10 Can Sealer

6.11 Cans and lids (commonly referred to as tuna cans)

6.12 8 oz, straight sided polypropylene jars or equivalent; (used for 25 mL and 100 mL geometries)

6.13 Teflon® or glass beakers (250 mL, 400 mL)

6.14 Disposable digestion vessels

6.15 Muffle furnace (programmable)

6.16 TEXPEN®

6.17 Teflon® beaker covers

6.18 Watch glasses

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 DI Water

7.3 Nitric acid (16 M HNO_3) concentrated

7.3.1 Nitric acid (4 M HNO_3) – to an appropriately sized bottle containing 1500 mL of DI water; add 500 mL of 16 M HNO_3.

7.4 Hydrochloric acid (12 M HCL) – concentrated, 37.2%

7.5 Hydrofluoric acid (HF 48.52%) – concentrated

7.6 Radiacwash™ solution 10% - add 100 mL of Radiac to 1 L of water

7.7 Bleach solution 10% - add 100 mL of bleach to 1 L of water

7.8 Sodium Sulfate

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2, unless I-129 or I-131 is requested. Samples collected for I-129 or I-131 analysis are ***not preserved***.

8.3.1 The pH of aqueous samples is checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis

8.3.1.1 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior to analysis.

8.4 Milk samples are not chemically preserved.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process. Where no preparation method exists (i.e. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (DU).

9.2 **Method Blank (MB)**

9.2.1 The method blank is in the same geometry as the majority of the samples in the batch.

9.2.2 The Method Blank is a pre-prepared empty geometry that is analyzed with every batch.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 The LCS is in the same geometry as the majority of the samples in the batch.

9.3.2 An LCS is a blank matrix spiked with a known amount of analyte(s).

9.3.3 The LCS is either a purchased sealed source standard or is made in-house.

9.3.4 An LCS is in a pre-prepared container that is analyzed with every batch.

9.4 **Sample Duplicate (DU)**

9.4.1 A replicate analysis of the original sample counted on a different detector will be performed as the duplicate.

9.5 **Procedural Variations/ Nonconformance and Corrective Action**

9.5.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6 **Decontamination of Tuna Can Sealer**

9.6.1 The sealer must be wiped down with 10% Radiacwash™ solution daily

9.6.1.1 Analyst must record the information in the daily logbook..

9.6.2 Sealer will be monitored for contamination as part of the monthly contamination survey, as per SOP ST-RP-0032

10.0 CALIBRATION AND STANDARDIZATION

10.1 The balance must be calibrated in accordance with ST-QA-0005.

10.2 For Gamma Spectroscopy calibration requirements, see ST-RD-0102.

11.0 PROCEDURE

11.1 See Attachment 2 for Automatic Canning Instructions

11.2 Liquid Sample Preparation

11.2.1 Liquid samples shall be prepared as a 25 mL, 100 mL, 500 mL, or 1L geometry.

11.2.2 Determine the proper geometry.

11.2.2.1 The volume of sample used depends on the amount required to meet the detection limits, the volume of sample supplied by the client, and whether the sample has very high activity. The sample volume may be reduced for high activity samples due to detector dead time considerations. Consult the count room supervisor or

radiochemistry technical director, if the sample has high activity which may require such consideration.

11.2.3 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.

11.2.4 Write sample information (i.e. ID #) on the geometry.

11.2.5 Measure the required sample volume (25, 100, 500 or 1L) by comparing the sample to the reference geometry.

11.2.5.1 Reference geometries are pre-made geometries comprised of DI water measured volumetrically.

11.2.5.2 Ra-226 is best reported inferred from the Bi-214 daughter after a 21-day ingrowth to allow the Ra-226 progeny through the potentially volatile Rn-222 daughter to reach secular equilibrium. To ensure Rn-222 is not lost from the geometry or does not escape into the headspace of the geometry, a radium-specific geometry should be utilized (e.g. 500 mL or 1L Marinelli beaker) which contains minimal headspace and can be sealed to prevent loss of radon. If Ra-226 is reported inferred from Bi-214 without sufficient ingrowth or from a geometry which is not intended for this purpose (due possibly to insufficient sample available), a NCM should be written to be included in the case narrative of the report.

11.2.5.3 If the client does not provide sufficient sample, and the sample is near a larger geometry, rather than reducing the volume significantly it may be preferable to dilute an aqueous sample with DI water to the correct volume in order to achieve a lower MDC. Consult Supervisor/Manager to determine which action is preferable.

11.2.5.3.1 If the sample is diluted, the undiluted volume is recorded as the sample volume. The dilution is only for fitting the calibrated geometry.

11.2.6 Place the lid securely on the geometry.

11.2.6.1 Remove excess air from Marinelli.

11.2.6.2 If the density is suspected to be greater than 1.2 g/ mL or less than 0.98 g/mL, generate a NCM. To determine the density use form RAD-0075_Density.xls (include form with batch paper work if utilized) Attachment 1

11.2.7 Seal the lid using plastic electrical tape. Marinelli beakers are prone to leaking liquids; the tape is tightly wrapped around the lid and the beaker in three layers each overlapping the previous layer with half the width of the tape. Make sure there are no creases in the tape which will form a channel for leakage.

11.2.8 Inspect for leakage.

11.2.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.3 Soil Sample Preparation

11.3.1 Soil samples for I-129 or I-131 analysis **are not dried and ground** but rather inserted into an appropriate calibrated geometry. Proceed to step 11.3.3.

11.3.2 Soil samples, which do not require I-129 or I-131 analyses, are prepared in accordance with SOP ST-RC-0003.

11.3.3 Soil samples shall be prepared as 230 mL sealed (tuna) can, 25 mL or 100mL straight sided poly jar, or 500 mL Marinelli geometry based on the amount of available sample. An Air Filter Geometry may be used if less than 25 mL of sample is available. In both the tuna can and marn soil geometries, the soil should nearly fill the geometry.

11.3.3.1 For I-129 analysis only a 25 mL or 100 mL straight sided poly jar geometry may be used (check with count room analyst on which geometry I-129 is calibrated for and prep the sample using the appropriate geometry).

11.3.3.2 Ra-226 is best reported inferred from the Bi-214 daughter after a 21-day ingrowth to allow the Ra-226 progeny through the potentially volatile Rn-222 daughter to reach secular equilibrium. To ensure Rn-222 is not lost from the geometry or does not escape into the headspace of the geometry, a radium-

specific geometry should be utilized (e.g. tuna can or 500mL Marinelli beaker) which contains minimal headspace and can be sealed to prevent loss of radon. If Ra-226 is reported inferred from Bi-214 without sufficient ingrowth or from a geometry which is not intended for this purpose (due possibly to insufficient sample available), a NCM should be written to be included in the case narrative of the report.

11.3.4 Write sample information (i.e. ID #) on the sample geometry.

11.3.5 Pre-weigh the empty geometry (tare weight) and record weight in TALS.

11.3.6 Fill the geometry with the appropriate amount of sample as described below.

11.3.6.1 Fill tuna cans above the ridge mark with sample. If there is insufficient sample, reduce geometry size.

11.3.6.2 Fill 100 mL geometry to the appropriate level by comparing to the reference geometry. If there is insufficient sample to fill, reduce the geometry size.

11.3.6.2.1 A 100 mL reference geometry is filled to the appropriate volume.

11.3.6.3 Fill 25 mL geometry to the appropriate level by comparing to the reference geometry. If there is insufficient sample, consult your supervisor or manager., If approval is given, an air filter geometry may be selected.

11.3.6.3.1 A 25 mL reference geometry is filled to the appropriate volume.

11.3.6.4 If air filter geometry is approved for use, spread soil evenly in a thin layer on the bottom of the petri dish. Do not put soil on a planchette; it needs to sit directly in the petri dish.

11.3.6.5 Fill 500 mL Marinelli beakers to the ridge mark just below the lid with sample. If there is insufficient sample to fill the marn soil to the ridge, reduce geometry size.

11.3.7 Close the sample geometry securely.

11.3.8 For tuna cans, seal with can sealer, wipe samples clean with paper towel and DI water.

11.3.9 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of geometry plus sample, minus the tare weight of the empty geometry).

11.3.10 For sampless that are not in tuna cans, seal the lid tightly using plastic electrical tape.

11.3.11 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.4 Vegetation Sample Preparation (No digestion)

11.4.1 Vegetation samples may be prepared in an appropriate calibrated geometry counted directly as dried and chopped matrix or green unprocessed matrix (if directed to do so by the client or if I-131 or I-129 is to be reported).

11.4.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by client or Project Manager.

11.4.1.1.1 Vegetation samples **for I-129 or I-131 analysis are not dried** but rather inserted into an appropriate calibrated geometry.

11.4.1.1.2 Dry weight can be determined on sample(s) not dried by using the percent moisture.

11.4.1.2 Consult the client requirements, client requirement memorandums or the Supervisor/Manager to determine proper sample handling.

11.4.2 The sample shall be counted in a 500 mL Marinelli, 230 mL sealed (tuna can), or a 25 mL or 100mL straight sided poly jar. The geometry is filled to the appropriate level with the sample.

11.4.2.1 I-129 analysis uses only a 25 mL or 100 mL straight sided poly jar geometry (check with count room analyst on which geometry I-129/I-131 is calibrated for and prep the sample using the appropriate geometry).

11.4.2.2 Ra-226 is best reported inferred from the Bi-214 daughter after a 21-day ingrowth to allow the Ra-226 progeny through the potentially volatile Rn-222 daughter to reach secular equilibrium. To ensure Rn-222 is not lost from the geometry or does not escape into the headspace of the geometry, a radium-specific geometry should be utilized (e.g. tuna can or 500 mL Marinelli beaker)

which contains minimal headspace and can be sealed to prevent loss of radon. If Ra-226 is reported inferred from Bi-214 without sufficient ingrowth or from a geometry which is not intended for this purpose (due possibly to insufficient sample available), a NCM should be written to be included in the case narrative of the report.

11.4.3 Write sample information (i.e. ID #) on the geometry.
11.4.4 Pre-weigh the empty geometry and record weight in TALS.
11.4.5 Place sample in the tared geometry.
11.4.5.1 Compress the sample when filling a 500 mL Marinelli beaker
11.4.6 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of geometry plus sample minus the tare weight of the empty geometry).
11.4.6.1 Verify with the Project Manager if sample is to be reported on a wet or dry basis.
11.4.7 Close the sample geometry securely, seal with plastic electrical tape
11.4.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.5 Vegetation Sample Preparation (with digestion)
11.5.1 Vegetation samples may be digested and then placed into an appropriate calibrated geometry and counted as a liquid matrix.
11.5.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by the client or project manager.
11.5.1.1.1 **Iodine isotopes (e.g. I-125, I-129 or I-131) cannot be prepared using digestion technique** due to volatility.
11.5.1.2 Consult the client requirements and Supervisor/Manager to determine proper handling.
11.5.2 Consult your supervisor/manager if a vegetation sample requires digestion.
11.5.3 After sample digestion, label appropriate geometry and bring to volume to match the reference geometry.
11.5.4 Close the geometry securely and seal with plastic electrical tape.
11.5.5 Inspect for leakage.
11.5.6 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.6 Air Filters/Swipes (no digestion)
11.6.1 Air filters may be counted as single filters or as composite filters.
11.6.1.1 Consult client requirements and Supervisor/Manager for instruction.
11.6.1.2 Air filters are reported as pCi/sample or pCi/g. The weight of the air filter is required if the reporting units is pCi/g.
11.6.2 Write sample information (i.e. ID#) on Petri dish.
11.6.3 Filters with reporting units of:
11.6.3.1 pCi/g proceed to 11.6.4
11.6.3.2 pCi/sample proceed to 11.6.5
11.6.4 Pre-weigh empty Petri dish, record the weight in TALS and then on the lid of the Petri dish.
11.6.5 Load air filter(s) directly into Petri dish
11.6.6 Place lid on Petri dish, for pCi/g record the weight of the sample (geometry and sample weight, minus geometry weight) in TALS.
11.6.7 Secure the Petri dish lid with plastic electrical tape
11.6.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.7 Air Filters/Swipes (with digestion)
11.7.1 Air filters are digested and counted as a liquid.

11.7.2 Consult your supervisor/manager if a air filter or swipe sample requires digestion.After sample digestion. label appropriate geometry and bring to volume using reference geometry as a guide.
11.7.3 Place lid securely on the geometry.
11.7.4 Seal the lid using plastic electrical tape.
11.7.5 Inspect for leakage.
11.7.6 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.8 Core Samples
11.8.1 To obtain sample, cut Shelby tube or sample container into two pieces.
11.8.1.1 Using a rigid pipe cutter, cut the tube completely through.
11.8.1.2 Using a wire saw, cut through the sample.
11.8.1.3 Cuts should be made at 2 inch intervals.
11.8.1.4 Remove sample from every other sliced section of the Shelby tube.
11.8.1.5 Dry and grind the sample as described in SOP ST-RC-0003.
11.8.2 Aliquot 500 g of sample
11.8.2.1 If less than 500 g of sample is available, contact Supervisor/Manager for instruction.
11.8.2.2 Soil samples shall be prepared as 200 mL sealed (tuna) can, 100 mL, or 25 mL, or 500 mL Marinelli geometry based on the amount of available sample. In the tuna can geometry, the soil should nearly fill the geometry.
11.8.3 Pre-weigh the empty geometry and record the weight on the geometry lid and in TALS
11.8.4 Writer sample information (i.e. ID#) on the sample geometry
11.8.5 Place the dried sample into the geometry for counting.
11.8.6 Weigh and record sample weight in TALS.
11.8.7 Secure the lid on the geometry with plastic electrical tape.
11.8.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.9 Food: vegetables, produce, grain or animal feed:
11.9.1 Vegetables, produce and grain samples may be prepared in a 500 mL Marinelli beaker or 1 L Marinelli beaker geometry depending on the requested reporting limit and available sample volume. These matrices are counted directly as whole grain, chopped or blended produce or vegetable matrices **without drying** unless directed by the client to dry the matrix.
11.9.1.1 Consult the client requirements and Supervisor/Manager for instruction.
11.9.2 For vegetables and produce, prepare the sample by chopping with a knife on a cutting board or using a food processor.
11.9.3 Write sample information (i.e. ID #) on the appropriate geometry.
11.9.4 Pre-weigh the empty geometry and record weight on geometry and in TALS.
11.9.5 Place processed sample in the pre-weighed geometry.
11.9.6 Compress the sample when filling a 500 mL Marinelli beaker
11.9.7 Weigh sample and record the weight in TALS in grams.
11.9.8 Close the sample geometry securely, seal with plastic electrical tape.
11.9.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.10 Food: meat and fish:
11.10.1 Meat and fish may be prepared in tuna cans, an appropriately sized Marinelli beaker. or a 25 mL or 100 mL geometry depending on the requested reporting limit and available sample volume. These matrices are counted directly **without drying.**
11.10.1.1 Consult the client requirements and Supervisor/Manager for instruction.

11.10.2 For meat and edible portions of fish, prepare the sample by chopping with a knife on a cutting board.

11.10.2.1 Fish sample are to be filleted prior to chopping.

11.10.2.2 For analysis of fish **when the whole fish is required to be analyzed**, remove the head with a knife and cut the fish into pieces of appropriate size to easily fit into the Marinelli beaker without air voids. Place the heads in the middle portion of the Marinelli and surround it with pieces to eliminate air voids or spaces.

11.10.3 Write sample information (i.e. ID #) on the geometry.

11.10.4 Pre-weigh the empty geometry and record weight on geometry and in TALS.

11.10.5 Place processed sample in the pre-weighed geometry.

11.10.6 Compress the sample evacuating any space in the geometry when filling a Marinelli beaker

11.10.7 Weigh sample and record the weight in TALS in grams.

11.10.8 Close the sample geometry securely, seal with plastic electrical tape.

11.10.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.11 Store unused portions of sample in appropriately sized poly containers. Food and vegetation samples are to be refrigerated.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 There are no calculations pertaining to this sample preparation procedure.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

12.3 Percent Moisture: Mass of original sample minus mass of dried sample divided by mass of dried sample.

12.3.1 $$p = \frac{W - D}{D}$$

p = fraction of total evaporable moisture content of sample
W = mass of the original sample
D = mass of dried sample

12.4 Density: Sample weight divided by the volume of said sample weight

12.4.1 $$d = \frac{m}{v}$$

d = density
m = mass
v = volume

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM.

14.3 Training Qualification

14.3.1 The Supervisor/Manager has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in ST-QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where feasible, technological changes have been implemented minimizing the potential for pollution to the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

16.2.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method EPA 901.1.

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, GA-01-R.

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revision.

17.5 Associated SOPs

17.5.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
17.5.2 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil and Grease Samples for Radiochemical Analysis
17.5.3 ST-RD-0102, GammaVision Analysis
17.5.4 ST-RP-0032, Instrumentation and Surveillance
17.5.5 ST-PM-0002, Sample Receipt and Chain of Custody
17.5.6 ST-QA-0002, Standard and Reagent Preparation
17.5.7 ST-QA-0036, Non-conformance Memorandum (NCM) process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 No Changes, Annual Review
19.2 Rev 11:
- 19.2.1 Inserted instructions regarding requirements for checking the pH of samples upon receipt and or prior to analysis in section 8.3.1.
- 19.2.2 Updated when sample ID's should be written on the container in section 11.0.
- 19.2.3 Inserted instructions for generating container ID labels throughout section 11.0.
- 19.2.4 Updated when weighing and recording sample weights/mass should be documented in gamma worksheet in section 11.0.
- 19.2.5 Added instructions for properly cleaning tuna cans before storage in section 11.2.9.

19.3 Rev 12:
- 19.3.1 Extensive revision to entire procedure
- 19.3.2 Addition of attachment 1

19.4 Rev 13:
- 19.4.1 Added sodium sulfate to section 7.0.
- 19.4.2 Updated section 9.2 regarding the matrix of method blanks.

19.5 Rev 14:
- 19.5.1 Updated vegetation sample with and without digestion throughout section 11.3 and 11.4.

19.6 Rev. 15: (8/30/2013)
- 19.6.1 Grammatical corrections and removal of references to QuantIMS through out
- 19.6.2 Section 3, added definition of replicate analyses
- 19.6.3 Section 8, removal of 180 day holding time
- 19.6.4 Section 9, explained that replicate will be used as duplicate for this procedure
- 19.6.5 Section 9.6, added "record in daily logbook"
- 19.6.6 Deleted record tare weight on lid in section 11.1.2
- 19.6.7 Deleted record weight of sample (container and sample weight minus container weight) on lid in section 11.1.8 , 11.2.9, 11.3.6
- 19.6.8 Added record weight in TALS throughout SOP
- 19.6.9 Added print proper paperwork throughout SOP

19.7 Rev. 16: (01/16/2015)
- 19.7.1 Grammatical corrections through out
- 19.7.2 Updated section 11.0
- 19.7.3 Added SOP references to Section 17: ST-RC-0014, St-RP-0032

19.8 Annual Review – No Changes (01/26/2015)
19.9 Annual Review- minor, non-procedural updates made (12/20/2016)
19.10 Revision 17 (12/15/17 Tech Review M. Minier/T Romanko; QA Review M Ward)
- 19.10.1 Added information pertaining to prep for Radium-226 in Sections 11.1, 11.2 and 11.3

19.11 Revision 18 (12/3/2018) Tech Review – M. Aldridge; QA Review – K. Ely
- 19.11.1 Made minor corrections throughout
- 19.11.2 Removed references to SOP ST-RC-0014 (SOP was archived)
- 19.11.3 Change references from normality "N" to molarity "M"
- 19.11.4 Section 9:
  - 19.11.4.1 updated section to match current practice
  - 19.11.4.2 removed duplicate paragraph about NCM's
- 19.11.5 Section 11:
  - 19.11.5.1 Added reference to Attachment 2
  - 19.11.5.2 Removed the requirement to record weight on container lid (section 11.2.5 and 11.4.4)
  - 19.11.5.3 Updated/clarified geometries used
- 19.11.6 Added Attachment 2 – Automatic Canning Machine Instructions

19.12 Added Eurofins logo, updated copyright information (4/17/2019)
19.13 Revision 19 (11/12/2019) Tech Review- C. Mazariegos; QA Review- K. Ely
- 19.13.1 Grammatical corrections throughout
- 19.13.2 Removed procedures for performing digestions and replaced with instructions to consult a supervisor from sections 11.5 and 11.7

19.13.3 Updated verbiage regarding containers to geometries
19.13.4 Clarified 11.3.6.2 and 11.3.6.3 procedure to fill to reference geometry level, and added supervisor approval needed for geometry smaller than 25 mL d=1
19.13.5 Added 11.3.6.4 air filter geometry procedure instructions for soil samples
19.13.6 Updated section 9.2 to reflect current practice using an empty geometry for all matrices as the method blank
19.13.7 Updated SOP names in section 17
19.13.8 Updated Attachment 1

Attachment 1

Eurofins TestAmerica St. Louis

DENSITY CORRECTION

Digi Tube Lot #: _____

Date: _____ Balance ID: _____

Analyst: _____ Pipette ID: _____

	Sample ID:	Container Wt (g)	Sample Volume (mL)	Sample + Container Wt (g)	Density (g/mL)	Original Sample Aliquot (g)	Adjust Sample Aliquot (mL)
1							
2							
3							
4							
5							
6							

Instructions:

Record the weight of a Class A digi tube in "Container Wt(g)"; record the volume of the sample added to the digit tube in "Sample Volume (mL)"; record the sample and digi tube in "Sample + Container Wt(g)." "Original Sample Aliquot" is the full sample size used for the actual analysis.

This density is to be used for sample aliquot correction when a sample is suspected of density less than or greater than 1. Include this document with batch paperwork.

Review is to be completed by a peer (someone other than the analyst handling the samples).

Reviewed by: _____ Date: _____

Attachment 2

Automatic Canning Machine Instructions

OVERVIEW

1. These instructions apply to the “All American Manual Can Sealer” Model 225.
2. The machine seals a tuna can by operation of the crank. One arm (marked F for 'First') crimps the lid onto the can, and then the other arm (marked S for 'Second') shapes it correctly.
3. Currently a cordless drill is used instead of a manual crank handle.

OPERATION

1. Make sure the F arm will act first by turning the crank a little. If necessary, rotate the crank until the F arm is about to close on the can but has not yet done so.
2. Take the can with the lid on it, and set it on the round platform. Hold the lid down so you can guide the can to meet the round metal plate on top that holds down the lid, while turning the lever to raise the platform. This prevents misalignment.
3. Turn the crank shaft by operating the cordless drill. While pressing its switch with the right hand, hold its battery with the left hand to prevent the drill itself from rotating.
4. The F arm will crimp the lid, and then the S arm will shape the crimp. Continue rotating the crank until the S arm has finished and moved away from the can completely. Stop the drill.
5. Turn the lever to lower the platform. Remove the can.
6. If the crank shaft is turned in reverse, the platform rotates in reverse, lowering the can (often without the lid attached) and throwing the lever unexpectedly. This is undesirable.
7. The drill may come loose during operation. Tighten the keyless chuck regularly to prevent a loose connection and deformation of the crank shaft. When tightening take care to not deform the crank shaft.

MAINTENANCE

1. The following should be performed once a day during heavy usage, or once a week during light usage:
   A. There are 3 holes marked "OIL" along the top, on the left side. Put one drop of machine oil in each hole.
   B. There is 1 unmarked hole on the left side directly under the adjustment bracket and above the return spring for that arm. Put 3 drops of machine oil in that hole.
   C. The rotors on the chucks (marked F and S) each have a bolt, with a slot for a screwdriver. If you were to take out the bolt you would find that the slot continues down one side of the bolt, so that you may put a drop of oil on the slot and it will seep down into the raceway and lubricate the rotor. Put 1 drop of machine oil in each slot. Rotate to spread the oil.
   D. The roller on the adjustment bracket can use 1 drop of machine oil directly on the exposed portion, and/or 1 drop in the slot adjacent to its axis pin. Rotate to spread the oil.

2. The following should be performed every six months:
   A. Remove the gearbox cover on the left side by removing the left arm return spring, unscrewing the 5 cover bolts with a 5/32" hex wrench, then rotating the cover back and forth while pulling on it. Apply thick grease (the size of a gumball) on the top of the worm gear with a popsicle stick, and rotate the crankshaft to spread the grease all around the main gear. Repeat two more times. Also apply a fair amount of grease on the round axle anchor on the cover plate.
   B. Remove the gearbox cover on the front (marked "Automatic Master-Sealer") by inserting a thin object (i.e. a paperclip) into the hole and pulling it out. Apply a fair amount of grease all over the conical gear teeth with a popsicle stick. Reach all the way back on both sides. Put the cover back in carefully since it is very malleable tin. Do not hammer or hit. The sides may be flared out slightly if it is too loose.
   C. Turn the lever to raise the platform. Apply a thin film of grease on the twisting part at the bottom.

Attachment 2 (Continued)

D. Remove the rotors from the chucks and clean them, and the bolts, with solvent. Apply a thin film of grease with your finger on all parts that rub together including the bolt. Reassemble.

3. When cleaning the exterior of the machine with solvent, be careful not to allow the solvent to get into areas that should be lubricated. The solvent will quickly break down the oil or grease, leaving no lubrication.

CONFIGURATION

1. Each ARM ASSEMBLY consists of:
   A. A primary arm that holds a chuck (marked F or S) and its rotor.
   B. A secondary arm, marked CY-24, with an adjustment screw and nut.
   C. An adjustment bracket, marked CY-22, with a roller.

2. The chucks (marked F or S) may be swapped between arms, since the parts are symmetrical. Whichever arm holds the F is to be used first. Whether that is on the left arm or right arm does not matter, as long as the operator uses the F arm first. Currently the F is on the right arm.
3. The rotors on the F and S chucks are shaped differently, since they perform different functions. They are not interchangeable between chucks. Each one must use its own type of rotor (crimping vs. shaping). The rotor bolts are sized differently.
4. The adjustment screw and nut on the secondary arms (marked CY-24) affects the crimp (for the F arm) and the shaping of the crimp (for the S arm). See the ADJUSTMENT section below.
5. The adjustment bracket has several holes marked 1, 2, 3, 2-1/2, and S. These are can sizes and do not need to be changed since only one size of can is used, the #2 which it is currently set to.

ADJUSTMENT

1. Loosen the nut on the adjustment screw on each arm. Back out the screw 5-7 turns so that the rollers will not contact the lid when the arms are in the closed position.
2. Put a can with lid on the platform. Raise the can into position.
3. Rotate the crank until the F arm closes completely, although the roller should not be touching the lid.
4. Tighten the adjustment screw until the roller touches the lid. Continue until the slot of the screw is vertical.
5. Write down "0" on a piece of paper. This will keep track of the position of the screw.
   A. Tighten the adjustment screw 1/4 turn and write down "1/4" on the paper.
   B. Rotate the crank until the F arm has finished crimping the can, moves away, and the S arm starts to move.
   C. The S arm should not touch the can, since the adjustment screw is backed all the way out. Continue rotating the crank to allow the S arm to complete its cycle.
   D. Inspect the crimp. If it looks correct then continue; otherwise put the can back and repeat steps A-C above.
   E. If you went too far, resulting in an unsatisfactory crimp, back off the adjustment screw 1/2 turn and try again with a new can.

6. Repeat the same steps for the S arm. This shaping process will require a can that has been already crimped, so that is why the F arm was adjusted first. It is advisable to crimp it once and remove the can between S arm adjustments to avoid deformation from repetition.
7. When both arms have been adjusted, hold the adjustment screw in position with a screwdriver while tightening the nut with a wrench. Do not over tighten the nut.
8. Default settings:
   A. F arm, 3/4 turn from first contact with a fresh lid.
   B. S arm, 1/2 turn from first contact with a crimped lid.

Title: RADIUM-226 AND RADIUM-228 BY CHEMICAL SEPARATION PREPARATION

Approvals (Signature/Date):

Chelsea Mazariegos 03/05/2019
Chelsea Mazariegos Date
Radiochemistry Prep Supervisor

Michael Ridenhower 3/5/19
Michael Ridenhower Date
Health & Safety Manager / Coordinator

Kristen Ely 3/6/19
Kristen Ely Date
Quality Assurance Manager

Andrew Buettner 3-5-19
Andrew Buettner Date
Operations Manager

This SOP was previously identified as SOP No. ST-RC-0041 Rev. 17

1.0 SCOPE AND APPLICATION

Facility Distribution No.: 0	Distributed To: See Electronic Distribution Sheet

1.1 This method covers the chemical separation preparation for radium-226 and radium-228 by direct measurement of its beta emitting progeny, Actinium (^{228}Ac). It is applicable to liquid (e.g water and wastewater) where complete dissolution and carrier exchange are readily achievable in the laboratory. For media where chemical dissolution is impractical, non-destructive measurement of the three principal photons of ^{228}Ac by gamma spectrometry is better suited.

1.2 This SOP is based on EPA Method 904.0, 903.0, SW9315, and SW9320.

1.3 The barium sulfate precipitate from this procedure contains all radium isotopes.

1.4 The barium sulfate can be counted for total alpha radiation. The time of the last barium sulfate precipitation should be recorded and used in calculating the in-growth factor.

1.5 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Radium isotopes are collected by coprecipitation with barium and lead sulfate and purified by precipitation from EDTA solution. After a suitable ingrowth period, ^{228}Ac is separated and carried on yttrium oxalate, purified and counted for the presence of total beta radiation. The precipitation and counting are performed in a manner consistent with the time requirements of the 6.13 hour half life of ^{228}Ac. By applying correction factors for ingrowth and decay and appropriately calibrating the beta counter, radium-228 is quantified. The barium sulfate fraction, minus radium-224, is counted by Gas Flow Proportional Counting (GFPC) to report radium-226.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 Strontium 90 or other beta emitting radionuclides that are carried by the yttrium oxalate precipitate (i.e. certain mixed fission or activation products) will yield a positive bias to the radium-228 values.

4.2 Samples which contain natural barium cause inaccurate chemical yield determinations.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [(1)]	Hazards	Exposure Limit [(2)]	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Acetic Acid, Glacial	Corrosive Flammable	10 ppm (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm- (TWA) 4 ppm- (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M^3- (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Centrifuge tubes, 50-mL

6.2 Centrifuge

6.3 Hot Plate

6.4 Analytical balance (four decimal places)

6.5 Stainless steel planchets

6.6 Beakers, various volumes

6.7 Syringe, 20-mL, 50 mL, 60 mL

6.8 Water Bath

6.9 Desiccator

6.10 Pipette

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI water

7.3 Acetic acid, 17.4 M: glacial CH_3COOH (concentrated), specific gravity 1.05, 99.8%.

7.4 Ammonium hydroxide, 15 M: NH_4OH (concentrated), sp. gr. 0.90, 56.6%.

7.5 Ammonium oxalate, 5%: non-critical reagent Dissolve approximately 2 g $(NH_4)_2C_2O \cdot H_2O$ in water and dilute to approximately 40 mL.

7.6 Ammonium sulfate, 200 mg/mL: non-critical reagent: dissolve approximately 400 g $(NH_4)_2SO_4$ in water and dilute to approximately 2000 mL.

7.7 Ammonium sulfide, 2%: Dilute 5 mL $(NH_4)_2S$, (20-24%), to 50 mL water; total volume 50 mL

7.8 CPI Barium carrier (standardized), 33.9 mg/mL, BaSO4 (20 mg/ml Ba)

7.8.1 If the barium carrier is not already standardized(from CPI) , standardize the barium carrier solution using the following procedure.

7.8.2 Pipette 1.0 mL barium carrier solution (20 mg/mL, Ba) into six separate labeled centrifuge tubes containing 15 mL DI H_2O.

7.8.3 To each tube, add 1 mL 18N sulfuric acid while stirring and digest precipitate in a hot water bath for approximately 10 min.

7.8.4 Cool, centrifuge and decant the supernate into appropriate waste container.

7.8.5 Wash precipitate with 15 mL DI water, centrifuge and decant the supernate.

7.8.6 Transfer the precipitate to a pre-weighed stainless steel planchet with a minimal amount of DI water.

7.8.7 Dry on a heat source. Store in desiccator until cool and weigh as barium sulfate.

7.8.7.1 Record the net weights of the precipitates and calculations in the Rad Standards Preparation Log.

7.9 Citric acid, 1M: non-critical reagent: Dissolve approximately 192 g $C_6H_8O_7 \cdot H_2O$ in water and dilute to approximately 1000 mL.

7.10 EDTA reagent basic (0.25M)non-critical reagent - dissolve approximately 20 g NaOH in approximataely 750 mL water, heat and slowly add approximately 93 g [ethylenedinitrilo] tetraacetic disodium salt, $(C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O)$ while stirring. Dilute to approximately 1 L.

7.11 Lead carrier, 15 mg/mL:non-critical reagent: Dissolve approximately 23.97 g $Pb(NO_3)_2$ in water, add approximately 5 mL of16M HNO_3 and dilute to approximately 1000 mL with water.

7.12 Lead carrier, 1.5 mg/mL:non-critical reagent: Dilute approximately 10 mL lead carrier, (15 mg/mL), to approximately 100 mL with water.

7.13 Methyl orange indicator, 0.1%:non-critical reagent: Dissolve approximately 0.1g methyl orange indicator in approximately 100 mL water.

7.14 Nitric acid, 16M: HNO_3 (concentrated), specific gravity 1.42, 70.4%.

7.14.1 Nitric acid, 6M: non-critical reagent: Mix approximately 3 volumes 16M HNO_3 (concentrated) with approximately 5 volumes of water.

7.14.1.1 Nitric acid, 2N: non-critical reagent: Mix approximately 1 volume 6N HNO_3 with approximately 2 volumes of water.

7.15 Sodium hydroxide, 18N:non-critical reagent:Dissolve approximately 72g NaOH in water and dilute to approximately 100 mL.

7.16 Sodium hydroxide, 10N: non-critical reagent: dissolve approximately 40g NaOH in water and dilute to approximately 100 mL.

7.17 Strontium carrier, 10 mg/mL: non-critical reagent: Dissolve approximately 24.16 g $Sr(NO_3)_2$ in water and dilute to approximately 1 liter.

7.18 Sulfuric acid, 18 N:non-critical reagent: cautiously mix approximately 1L36N H2SO4 (concentrated) with approximately 1L of water

7.19 Yttrium carrier(standardized), 5 mg/ml

7.19.1 Yttrium carrier from CBI- 50 mg/mL

7.19.1.1 If the yttrium carrier is not already standardized, standardize the yttrium carrier solution using the following procedure.

7.19.1.2 Prepare 10 samples of the carrier solution. Perform the following steps on each vial.

7.19.1.3 Add 5 mL DI water to a 50 mL centrifuge tube.

7.19.1.4 Add 2 mL concentrated nitric acid.

7.19.1.5 Add 1 drop of MCP indicator (0.1%).

7.19.1.6 Add 1.0 mL of yttrium carrier (10 mg/mL, Y) pipette.

7.19.1.7 Add 5 mL of 0.4 M oxalic acid.

CAREFULLY add concentrated ammonium hydroxide dropwise until a reddish yellow end point is obtained.

NOTE: The pH must be between 1.7 - 1.9 to assure a uniform $9{\cdot}H_2O$ hydrate yttrium oxalate precipitate. The yttrium oxalate will have precipitated.

7.19.1.8 Heat the solution for approximately 5 –10 minutes in a hot water bath.

7.19.1.9 Cool the solution for approximately 5 – 10 minutes in an ice water bath.

7.19.1.10 Centrifuge.

7.19.1.11 Decant supernate into the appropriate waste container.

7.19.1.12 Add approximately 20 mL of DI water to the precipitate.

7.19.1.13 Vortex and centrifuge.

7.19.1.14 Decant supernate into the appropriate waste container.

7.19.1.15 Add approximately 20 mL of DI water to the precipitate.

7.19.1.16 Vortex and Centrifuge.

7.19.1.17 Decant supernate into the appropriate waste container.

7.19.1.18 Add approximately 5 mL of D.I. water, slurry the precipitate.
7.19.1.19 Pre-weigh a planchet and record the tare weight on the sample worksheet.
7.19.1.20 Transfer the slurry to the planchet.
7.19.1.21 Dry the planchet on a hotplate.
7.19.1.22 Remove from heat and cool to room temperature in a desiccator.
7.19.1.23 Weigh sample to determine yttrium yield.
7.19.1.24 Repeat heating and cooling in the desiccator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are ± 5% or less.
7.19.1.25 Record gross and final weights in LIMS. .

7.20 Yttrium carrier,-9 mg/mL: Dilute 50 mL yttrium carrier, to 100 mL with water.

7.21 Strontium-yttrium mixed carrier, 0.9 mg/mL Sr^{+2}; 0.9 mg/mL Y^{+3}:
7.21.1 Dissolve 2.175 g of $Sr(N0_3)_2$ in 200 mL of DI water. Add 90 mL of 50 mg/mL Y carrier. Dilute to final volume of 500 mL

7.22 Radium-226, standard 20-25 dpm

7.23 Radium-228, standard 20-25 dpm

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 **Batch**
9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch comprises of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.
9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
9.2.2 A method blank must be prepared with every sample batch.
9.2.3 For Liquid analyses, the method blank is comprised of DI water acidified with 2 mL of nitric acid.
9.2.4 For Soil analyses, the method blank is comprised of DI water acidified with 2 mL of nitric acid.

9.3 **Laboratory Control Sample**
9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.3.2 An LCS must be prepared with every sample batch.
9.3.3 For Liquid analyses, the LCS is comprised of DI water fortified with radium-226 and radium-228.
9.3.4 For Soil analyses, the LCS is comprised of radium-226 and radium-228.

9.4 **Matrix Spike/Matrix Spike Duplicate**
9.4.1 A Matrix Spike (MS) is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch. MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate**
9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of an LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**
9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and thermometer calibration must be checked daily when used. Refer to SOP ST-QA-0005.

10.2 See the analytical SOP for instrument calibration; ST-RD-0403

11.0 PROCEDURE

11.1 Water Samples
11.1.1 Ensure that the sample container is capped tightly and shake it thoroughly. Ensure that the sample is at or below a pH of 2. Transfer a sample aliquot to a beaker.
11.1.2 Sample aliquot size is 1 liter.

11.1.2.1 For client requesting a reporting limit less than 1pCi/L, a larger sample volume may be required. Contact Radiochemistry manager/supervisor for instruction.

11.1.2.2 If less than 1 liter of sample was provided by the client, write the NCM noting insufficient volume.

11.2 Soil Samples

11.2.1 For soil samples prepare per SOP ST-RC-0003, "Drying and Grinding of Soil and Solid Samples", and weigh 1 to 2 grams into a labeled crucible.

11.2.2 Place in oven at 600 °C and allow to muffle for four hours. Allow to cool.

11.2.3 Transfer to digestion tube, or Teflon beaker using 4M HNO_3.

11.2.4 Add 1 mL of standardized barium carrier to samples and QC. Add radium-226 and radium-228 spike to LCS and MS/MSD, if applicable.

11.2.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.

11.2.6 Digest in mod block at >110°, or on a hotplate for four hours or until dry.

11.2.7 Carefully add 10 mL concentrated nitric acid, 10 mL concentrated hydrochloric acid and 5 mL concentrated Hydrofluoric acid.

11.2.8 Digest in mod block at >110°, or on a hotplate for four hours or until dry.

11.2.9 Dissolve with 10 mL HNO_3 and 10 mL HCl, return to mod block, or hotplate for 30min.

11.2.10 Transfer to 400 mL beakers with 4M HNO_3. Dilute to 200 mL with DI water.

11.3 Initial Precipitation

11.3.1 Add methyl orange indicator until pink endpoint persists. For soils skip to 11.3.4.

11.3.2 Add 1.0 mL standardized barium carrier (33.9 mg/mL).

11.3.3 Spike LCS and MS/MSD (if applicable) with radium-226 and radium-228.

11.3.4 Add 1M citric acid in ratio of 5 mL per liter. Mix thoroughly.

11.3.5 Add 2.5 mL lead carrier (15 mg/mL), 2 mL strontium carrier (10 mg/mL), and 0.2 mL yttrium carrier (9 mg/mL); stir well. (mass = 0.0249 g)

11.3.6 Slowly add 15M ammonium hydroxide until a definite yellow color is obtained, then add a few drops more. If no yellow endpoint is visible, insure pH is > 6.5.

11.3.7 Stir and heat until close to boiling for about 30 minutes.

11.3.8 **Face shield must be worn during this process.** Precipitate lead and barium sulfates by adding 10 mL of 18N sulfuric acid, or until the pink endpoint reappears.

11.3.9 Add 5 mL ammonium sulfate (200 mg/mL) for each liter of sample. Stir frequently and keep at a temperature of approximately 90°C for 30 minutes until a barium sulfate precipitate forms.

11.3.10 Allow precipitate to settle to the bottom of the beaker for a least 6 hours.

11.4 Reprecipitation Clean up / Ac-228 Ingrowth

11.4.1 Decant the supernate and discard to acid waste, taking care to avoid disturbing the precipitate.
Transfer precipitate to a 50 mL centrifuge tube, taking care to rinse last particles out of beaker with DI water. Centrifuge and discard supernate.

11.4.2 Wash the precipitate with 10 mL 16M HNO_3, vortex, centrifuge, and discard supernate to acid waste. Repeat this step.

11.4.3 Wash the precipitate with 10 mL D.I.H_2O, vortex, centrifuge, and discard supernate to acid waste. Repeat this step.

11.4.4 Add 1 mL strontium-yttrium mixed carrier if Ra-228 analysis is requested.

11.4.5 Add 30 mL basic EDTA reagent; vortex thoroughly, and heat in a hot water bath (approximately 80°C) until precipitate dissolves. There should be no precipitate remaining in the tube.

11.4.5.1 If insoluble solids remain in the tube after addition of EDTA, confirm that the pH is > 10.

11.4.5.2 If pH > 10, centrifuge and syringe filter the supernate into a clean, labeled 50 mL centrifuge tube. Discard insoluble residue.

11.4.6 Add 1 mL ammonium sulfate (200mg/mL) .

11.4.7 Add 2 mL of 17.4M acetic acid or until barium sulfate precipitates and vortex thoroughly.

11.4.8 Digest in a hot water bath until precipitate settles.

11.4.9 Centrifuge and discard supernate to acid waste..

11.4.10 Add 30 mL basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.

11.4.11 Add 1 mL ammonium sulfate (200mg/mL).

11.4.12 Add 2 mL of 17.4M acetic acid or until barium sulfate precipitates and vortex thoroughly.

11.4.13 Record the date and time of last barium sulfate precipitation (T1) . This is the beginning of the ^{228}Ac in-growth time.

11.4.14 Digest in a hot water bath until precipitate settles.

11.4.15 Centrifuge and discard supernate to ascid waste..

11.4.16 Add 20 mL basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.

11.4.17 Add 0.2 mL standardized yttrium carrier and 1 mL lead carrier (1.5 mg/mL).

11.4.17.1 If any precipitate forms, dissolve it by adding a few drops of 10N NaOH.

11.4.17.2 If radium-228 analysis is requested, cap the tube and allow it to age at least 36 hours.

11.4.17.3 If radium-226 analysis is requested, cap the tube and allow it to age (in-growth period is 14 or 21 days, see sample log in sheet).

11.5 Lead Scavenge Clean-up

11.5.1 Add 0.3 mL ammonium sulfide. Add 0.5 mL of 10N sodium hydroxide until lead sulfide precipitates, vortex than centrifuge.

11.5.2 Add 1 mL lead carrier (1.5 mg/mL), 0.1 mL ammonium sulfide, and 0.1 mL of 10N sodium hydroxide.

11.5.3 Centrifuge and filter supernate through 0.45 mm syringe filter into a clean labeled tube. Discard filter.

11.5.3.1 The half life ^{228}Ac is very short. Check with count room staff on scheduling of the GFPC. **Do NOT proceed with the remaining steps of this procedure until authorized by the count room GFPC analysts or Radiochemistry manager.**

11.6 Out of In-growth

11.6.1 Once yttrium hydroxide is precipitated, the analysis must be carried to completion to avoid excessive decay of ^{228}Ac.

11.6.2 Add 5 mL 18N sodium hydroxide, stir well and digest in a hot water bath, 70-85°C, until yttrium hydroxide coagulates, usually about 10 minutes. Centrifuge for 10 minutes and carefully decant supernate into a clean, labeled 50 mL centrifuge tube. Save for barium yield determination, Step 11.8.1

11.6.3 Record time (T2) of yttrium hydroxide precipitation; this is the end of the ^{228}Ac in-growth time and the beginning of ^{228}Ac decay time.

11.7 Yttrium Yield (for radium-228)

11.7.1 Weigh a stainless steel cleaned planchet. Record weight in the LIMS spreadsheet.

11.6.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.

11.7.2 Dissolve the precipitate from step 11.5.2 in 2 mL 6M nitric acid. Vortex and add 5 mL water and precipitate yttrium hydroxide with 3 mL 10N sodium hydroxide. Heat and stir in a hot water bath until precipitate coagulates. Carefully centrifuge for 10 minutes. Carefully discard supernate to base waste. (do not dump gel-like pellet).

11.7.3 Dissolve precipitate with 1 mL 2M nitric acid. Vortex.
11.7.3.1 If solution is still cloudy add 2M nitric acid drop wise until the solution clears.
11.7.4 Dilute to 5 mL with DI water and add 2 mL 5% ammonium oxalate. Centrifuge and discard supernate to acid waste.
11.7.5 Wash precipitate with 10 mL DI water.
11.7.6 Vortex, centrifuge and discard supernate to acid waste.
11.7.7 Plating
11.7.7.1 To determine yttrium yield, quantitatively transfer the precipitate to the previously weighed stainless steel cleaned planchet using a minimal amount of water.
11.7.7.2 Dry on a hot plate.
11.7.7.3 Upon dryness, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.
11.7.7.4 Record the weight in the LIMS worksheet to determine the chemical yield of the yttrium carrier solution.

11.8 Barium Yield (for radium-226)
11.8.1 Weigh a stainless steel cleaned planchet. Record weight in the LIMS spreadsheet.
11.8.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.
11.8.2 To the supernate from Step 11.5.2, add 5 mL 16M nitric acid and 2 mL ammonium sulfate (200mg/mL),. Add 5 mL 17.4M acetic acid until barium sulfate precipitates. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate to acid waste.
11.8.3 Add 30 mL basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitates does not readily dissolve.
11.8.4 Add 1 mL ammonium sulfate (200 mg/mL). Add 2 mL 17.4M acetic acid until barium sulfate precipitates and vortex thoroughly.
11.8.5 Record date and time (T3) of $BaSO_4$ precipitate in the LIMS data sheet.
11.8.6 Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate to acid waste.
11.8.7 Wash precipitate with 10 mL water. Vortex, centrifuge and discard supernate acid waste.

11.8.8 Plating
11.8.8.1 Transfer the precipitate to a pre-weighed stainless steel cleaned planchet with a minimal amount of water.
11.8.8.2 Heat the planchet again using the hot plate, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.
11.8.8.3 Record the final weight of the planchet in LIMS to determine the chemical recovery for the barium carrier solution.

11.9 Submit the planchets to the counting room for analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. LCS % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 There are no calculations pertaining to this sample preparation procedure.

12.3 Radium-226 and radium-228 by GFPC calculations are given in ST-QAM.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type “A” or “B”.

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid Rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0, Radium-228 in Drinking Water

17.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0, Alpha-Emitting Radium Isotopes in Drinking Water

17.3 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 9315, Alpha Emitting Radium Isotopes

17.4 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 9320, Radium-228

17.5 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.6 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revision

17.7 Associated SOPs:
- 17.7.1 ST-PM-0002, Sample receipt and Chain of Custody
- 17.7.2 ST-QA-0002, Standard and Reagent Preparation
- 17.7.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
- 17.7.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
- 17.7.5 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis
- 17.7.6 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
- 17.7.7 ST-RD-0403, Gas Flow Proportional Counting (GFPC) Analysis

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 After initial precipitation, TestAmercica St. Louis decants the supernate after the precipitate has been allowed to settle for at least six hours, as opposed to the EPA Method 904 which requires filtration to isolate the precipitate.

18.2 At the point of in-growth of actinium-228, TestAmerica St. Louis utilizes either a 14 days or 21 day in-growth depending on the client's request for wastewater and 21 days for drinking water before finishing the procedure.

18.3 TestAmerica St. Louis counts the barium sulfate fraction (minus the Ra-224) by GFPC to report radium-226; an option documented in section 10.5 of the EPA Method 903.0.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Annual Review, No Changes.

19.2 Rev. 10:
- 19.2.1 Updated section 11.4 to add carrier prior to adding EDTA.
- 19.2.2 Moved 11.4.15, recording the date/time of the last barium sulfide precipitation to section 11.4.9.

19.3 Rev. 11:
- 19.3.1 Updated soil LCS composition in section 9.3.4.
- 19.3.2 Updated amount of standardized yttrium carrier used in section 11.4.17
- 19.3.3 Updated amount of DI water for precipitate wash and removed repetition of wash and vortex steps in section 11.7.7 and 11.7.8.

19.4 Rev. 12:
- 19.4.1 Grammatical corrections throughout
- 19.4.2 RadCap/RadCapture changed to LIMS.
- 19.4.3 Deleted stir thoroughly

19.4.4 Section 11.3.3 changed time from 30 minutes to 5-10 minutes.
19.4.5 Section 11.3.5 added: add 10 mL, and deleted add 0.25 mL more.
19.4.6 Section 11.4.5.2 added syringe filter supernate into clean labeled tube.
19.4.7 Section 11.4.7 changed aliquot to 3 mL of acetic acid.
19.4.8 Section 11.4.9 moved to after 11.4.13.
19.4.9 Section 11.4.13 changed aliquot of acetic acid to 3 mL.
19.4.10 Section 11.5.1 removed stir well after ammonium sulfide is added, and changed aliquot of sodium hydroxide to 0.5 mL.
19.4.11 Section 11.5.2 changed aliquot of sodium hydroxide to 0.1 mL.
19.4.12 Section 11.8.2 deleted stir well after each addition and changed aliquot to 7 mL acetic acid.
19.4.13 Section 11.8.4 changed aliquot of acetic acid to 3 mL.
19.4.14 Updated section 15

19.5 Revision 13: (07/31/2014)
19.5.1 Section 7.8 added the name of the reagent, $BaSO_4$
19.5.2 Section 7.8.2 changed 16 to 20 mg/ml, Ba per certificate
19.5.3 Section 7.17 made correction for the concentration , replaced 9 with 10 mg/ml per method.
19.5.4 Section 7.19 added concentration, 5 mg/ml
19.5.5 Section 7.2 Corrected concentration from 9 to 2.5 mg/ml.

19.6 Revision 14: (08/11/2015)
19.6.1 Updated section 7.0
19.6.2 Updated section 11.0

19.7 Revision 15: (06/30/2016)
19.7.1 Updated section 11.0
19.7.2 section 11.4.5, 11.4.10, and 11.8.3, the amount of EDTA was increased from 20 mL to 30 mL.
19.7.3 section 11.4.6, 11.4.7, 11.4.11, 11.4.12, and 11.8.4, the amount of ammonium sulfate and acetic acid added was changed.
19.7.4 Section 11.6.2 the amount of sodium hydroxide and the time was updated. Recording the time of the yttirium hydroxide precipitation was moved to this step

19.8 Revision 16 (06/30/17)
19.8.1 Updated section 5.0- changed from MSDS to SDS
19.8.2 Updated section 7.0- clarified non-critical reagents, changed concentrations of reagents, removed “from Milli Q Unit”
19.8.3 Updated section 11.0- updated procedure- centrifuge times, where to dump supernate, added information about T-times

19.9 Revision 17 (05/17/2018)
19.9.1 Technical Review: S.Bernsen /QA Review M Ward
19.9.2 Updated section 6.6- added additional syringe volumes, 50 mL and 60 mL; removed 0.45mm filter

19.10 Revision 18 (03/08/2019)
19.10.1 Technical Review: C. Mazariegos/QA M Ward
19.10.2 Updated section 11.0
19.10.2.1 Section 11.3.1 changed “red color” to “pink endpoint” and added “for soils skip to 11.3.4.”
19.10.2.2 Section 11.3.2-3 moved spiking and tracing prior to adding any reagents that could cause a precipitation.
19.10.2.3 Section 11.3.6 added “slowly add” and “If no yellow endpoint is visible, insure pH is > 6.5.”
19.10.2.4 Section11.3.7 updated to “stir then heat until close to boiling for about 30 minutes.”
19.10.2.5 Section 11.3.8 changed “red color” to “pink endpoint”.
19.10.2.6 Section 11.3.9 added “until a barium sulfate precipitate forms.

19.11 Added Eurofins logo and updated copyright information (4/19/2019)

Title: GAMMAVISION® ANALYSIS

Approvals (Signature/Date):

Jody Watson 11-8-19
Jody Watson Date
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This SOP was previously identified as SOP No. ST-RD-0102 Rev. 18

Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1 This procedure applies to all germanium detectors and the computer assisted germanium spectroscopy analysis system.

1.2 Due to the nature of gamma spectroscopy, once the system is calibrated to a particular geometry a similar matrix may be run as long as it is prepared to match a calibrated geometry.

1.3 This SOP is based on EPA Method 901.1, DOE EML HASL 300 Method GA-01-R and ANSI N42.14-1999.

1.4 The requested limits (**RL**), minimum detectable amount (**MDA**) and QC limits are maintained in the Laboratory Information Management System (**LIMS**).

2.0 SUMMARY OF METHOD

2.1 This procedure provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the germanium spectroscopy system.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Germanium spectrometry has potential interference. Interferences are usually in the form of radionuclides with unresolved photon emissions. These interferences are limited by the careful design/construction of the gamma spectral identification and interference libraries.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6.0 EQUIPMENT AND SUPPLIES

6.1 Germanium spectroscopy system utilizing a computer based data acquisition system (GammaVision®-32).

6.2 GammaVision®-32 (know as GammaVision) is a comprehensive, all-in-one package, for the analysis of gamma-ray spectra acquired with HPGe detectors.

6.2.1 See “Maintenance Tracker” database for software version.

6.3 Global Value software is an optimization tool for automation, custom reporting, quality assurance and data management (GammaVision productivity add-on software).

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 Commercially prepared mixed gamma standards in reproducible geometries, with all appropriate NIST Source Certificate information.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Samples may be collected in glass or plastic containers.

8.2 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 See gamma preparation SOP (ST-RC-0025) for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample, and Sample Duplicate.

9.3 **Method Blank (MB)**

9.3.1 A method blank must be counted with every sample batch.

9.3.2 The method blank is in the same geometry as the majority of the samples in the batch.

9.3.3 The method blank is a pre-prepared empty geometry that is analyzed with every batch.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 A LCS must be counted with every sample batch.

9.4.2 The LCS is in the same geometry as the majority of the samples in the batch

9.4.3 An LCS is a blank matrix spiked with a known amount of analyte(s).

9.4.4 The LCS is either a purchased sealed source standard or is made in-house.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is a recounted field sample to demonstrate instrument precision, since there is no sample preparation (required to count on a different detector than the sample).

9.5.1.1 If requested, the laboratory may perform a Sample Duplicate which is an additional aliquot of a field sample.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Except in specific instances, it is NOT acceptable to remove points from a calibration curve. Points must never be removed solely for the purpose of meeting criteria. There must be a demonstrable reason to remove a point. Consult with the Supervisor or Technical Director for guidance. Examples of acceptable reasons for the removal of points include, but are not limited to:

10.1.1 A nuclide has decayed below acceptable count criteria due to short half-life

10.1.2 There is an apparent nuclide-specific issue associated with the purchased standard (e.g. all energy lines for a particular nuclide are low or high).

10.2 There are two types of Calibrations performed for Gamma: Energy and Efficiency

10.2.1 Energy Calibrations

10.2.1.1 Frequency: the energy calibration is performed once per detector . The source is not geometry specific.

10.2.1.2 A new calibration curve must be generated prior to initial use, following repair or replacement of a key detector part when subsequent performance checks indicate a change in performance, after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in instrument response, or when indicated by corrective actions.

10.2.1.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to Section 10.1.

10.2.1.4 Range: the energy range is determined by the sample matrix (e.g. 46.54 to 1836.1 keV).

10.2.1.5 Criteria:

10.2.1.5.1 The curve should have, at minimum, eight calibration points used to determine the energy relationship of the calibration.

10.2.1.5.2 The energy difference (delta Δ) should be within 0.05% for all calibration points or within 0.2 keV for the calibration points.

10.2.1.5.3 The FWHM must be less than 3.0 keV at 1332 keV.

10.2.1.5.4 FWHM difference (delta Δ) should be within 8% for all calibration points.

10.2.2 Efficiency Calibrations

10.2.2.1 Frequency: the efficiency calibration is performed per geometry.

10.2.2.2 A new calibration curve must be generated prior to initial use, following repair or replacement of a key detector part when subsequent performance checks indicate a change in performance, after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in instrument response, or when indicated by corrective actions.

10.2.2.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to Section 10.1.

10.2.2.4 Range: the energy range of the calibration is determined by the sample matrix e.g.. 46.54 to 1836.1 keV.

10.2.2.5 Broad-Range Energy/Efficiency Curve Criteria:

10.2.2.5.1 The curve should have at least eight points to determine the efficiency.

10.2.2.5.2 The calibration source must have radionuclides that “bracket” the intended range of calibration.

10.2.2.5.3 A minimum of 10,000 counts will be accumulated for each data point.

10.2.2.5.4 The efficiency difference (delta Δ) should be within 8% of the true value for each point, except when utilizing the True Coincidence Correction (TCC) calibration. In the TCC calibration, the nuclides subjected to significant coincidence

events (e.g. Cs-134, Y-88) will appear to be biased low and the lowest energy point (e.g. Pb-210 at 46.5 keV) may have greater variance. This is to be expected, and the apparent bias is corrected in the samples for these nuclides through the TCC correction function of the software. After reprocessing the calibration count using the new TCC calibration, all points should fall within 8% of the true/expected value for each point.

10.3 Initial Calibration Verification (ICV) [Frequency: Once]

10.3.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.3.1.1 The ICV check does not include short half-life nuclides which may exist in the purchased standard. At a minimum, the ICV will always contain Am-241 (low), Cs-137 (medium) and Co-60 (high).

10.3.2 An ICV must be performed with every initial calibration.

10.3.3 The ICV percent recovery must be within ± 10% of the true value for each nuclide.

10.3.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.4 Daily Checks

10.4.1 The detector **background** shall be checked each day that the germanium spectroscopy system is used. Limits are set at 2 sigma and 3 sigma.

10.4.1.1 Bkgd Countrate (background count rate for entire spectrum)

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.4.2 The instrument **Channel**, **Energy**, **FWHM** (resolution) and **Activity Difference** (efficiency) for a detector shall be checked each day the germanium spectroscopy system is used (using a check source that is non-geometry specific).

10.4.2.1 **Channel** – (low and high energy) is monitored for channel alignment. Limits are set around the target Channel.

10.4.2.1.1	QA-60	Low Energy		
		Tolerance (warning)	=	± 1 channel
		Control (out)	=	± 2 channels
10.4.2.1.2	QA-1332	High Energy		
		Tolerance (warning)	=	± 2 channels
		Control (out)	=	± 3 channels

10.4.2.2 **Energy** – (low and high energy) is monitored for energy alignment. Limits are set around a target energy.

10.4.2.2.1	QA-60	Low Energy		
		Tolerance (warning)	=	± 0.25 keV
		Control (out)	=	± 0.50 keV
10.4.2.2.2	QA-1332	High Energy		
		Tolerance (warning)	=	± 0.5 keV
		Control (out)	=	± 0.75 keV

10.4.2.3 Full-Width at the Half Maximum (**FWHM**) - (low, mid, and high energy) is monitored for peak shape There are no limits compared to a target FWHM. There are no lower limits (–) set for FWHM.

10.4.2.3.1	QA-60	Low Energy		
		Tolerance (warning)	=	+ 1.1
		Control (out)	=	+ 1.2
10.4.2.3.2	QA-662	Mid Energy		
		Tolerance (warning)	=	+ 1.7
		Control (out)	=	+ 1.8

10.4.2.3.3 QA-1332 High Energy

Tolerance (warning)	=	+ 2.2
Control (out)	=	+ 2.3

10.4.2.4 **Activity Difference** (low, mid, and high energy) – is monitored to check the percent difference between the source activity and the reported activity. Limits are set around the target activity.

10.4.2.4.1 QA-60/662/1332 Low/Mid/High Energy

Tolerance (warning)	=	± 4
Control (out)	=	± 5

10.4.3 If the daily check is outside of the control limits, it may be recounted (QSM and other project-specific quality plans may require two consecutive passing source counts before the detector can be used to count project samples).

10.4.3.1 If the out of control parameter is found acceptable for the rerun(s), the instrument can be used for the analysis of samples. ***Note***: *No corrective action is necessary for this situation since the uncertainty can be attributed to the stochastic uncertainty of decay process (statistics), uncertainty of the sources, or a known uncorrected trend.*

10.4.3.2 If the instrument fails to meet the acceptance criteria for the rerun, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out". (See ST-QA-0036 for NCM details regarding tagging out of service).

10.4.3.3 If the QC check fails the following day for the same detector for the same specific parameter as the day before, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out" until the detector can be evaluated and/or maintenance can be performed.

10.4.3.4 The analyst may want to:

10.4.3.4.1 Check the expiration date of the radioactive standard to confirm the material is current, for the isotopes being utilized.

10.4.3.4.2 Check source positioning and all instrument settings.

10.4.3.4.3 Check all cables for any apparent damage and confirm that all cables are routed to proper connectors and are in good working order.

10.4.3.4.4 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Corrective actions must be noted in the instrument maintenance log.

10.4.3.4.5 If a parameter has two successive values in the warning limits, the system will be examined for a trend and noted in the maintenance log. Decisions will be based upon the Data Quality Objectives (DQO) and the degree of the bias in relation to the parameter.

10.5 Background

10.5.1 Background subtraction spectrum shall be established for the germanium spectroscopy systems **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements.

10.5.1.1 Backgrounds count for a minimum of 12 hours.

10.5.1.1.1 If a client project requires a longer count time, then the background must be performed at the longer time before initiating analysis.

10.5.1.2 Monthly Background limits are set at 2 sigma and 3 sigma.

10.5.1.2.1 Bkgd Countrate (background count rate for entire spectrum)

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.6 Calibration Software Handling

10.6.1 Gamma Detector System Energy and Shape Calibration

10.6.1.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:
- 10.6.1.1.1 X = Detector Number
- 10.6.1.1.2 Analysis method
- 10.6.1.1.3 Select library
- 10.6.1.1.4 Enter correct sample data.
- 10.6.1.1.5 Enter correct conversion time.

10.6.1.2 Close all detectors windows in the current instance of gamma vision, then recall the appropriate calibration spectrum into the buffer window.
10.6.1.3 Select the menu "Analyze\Setting\Sample type…."
10.6.1.4 Select the browse button next to the "File" field and open the file. Click the "OK" button of the window to close it.
10.6.1.5 Recall the application Calibration File from the menu "Calibration \Recall Calibration…"
10.6.1.6 Select the menu "Calibrate\Calibration wizard…"
10.6.1.7 Select the option to create new energy calibrations. Select the next button.
10.6.1.8 On the energy calibration wizard page, select the file "DET_EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the next button.
10.6.1.9 Select the next button to perform the energy, FWHM.
10.6.1.10 Select the edit energy button to review the energy.
- 10.6.1.10.1 Close the energy calibration sidebar window.

10.6.1.11 Select the save calibration button and save the calibration to "Cal\Energy\X_Energy.clb" where X is the detector.
10.6.1.12 Enter the calibration description in the format "X_ENERGY_GEOMETRY" where X is the detector number and Geometry is an appropriate geometry description when prompted. Select the Finish button to close the calibration wizard.
10.6.1.13 Print the calibration report from the menu "Calibrate \print calibration.

10.6.2 Gamma Detector System Efficiency Calibration

10.6.2.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:
- 10.6.2.1.1 X = Detector Number
- 10.6.2.1.2 Analysis method
- 10.6.2.1.3 Select library
- 10.6.2.1.4 Enter correct sample data.
- 10.6.2.1.5 Enter correct conversion time.

10.6.2.2 Close all detector windows in the current instance of Gamma Vision, then recall the appropriate calibration spectrum into the buffer window.
10.6.2.3 Select the menu "Analyze\Setting\Sample Type".
10.6.2.4 Select the browse button next to the "File", field and open the file. Click the "OK" button at the bottom of the window to close it.
10.6.2.5 Recall the applicable calibration file from the menu "Calibration\Recall Calibration" (if the geometry file currently exists).
10.6.2.6 Select the menu "Calibrate\Calibration Wizard".
10.6.2.7 Select the option to create new energy and efficiency calibration. Select next button.
10.6.2.8 On the Energy Calibration Wizard page select the file "EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the Next button.
10.6.2.9 On the Efficiency Calibration Wizard page, select library file, "DET_EfficiencyCalibration.Lib" for mixed gamma sources.

10.6.2.10 On the Efficiency Calibration Wizard page, select the appropriate Certification file from the directory.
10.6.2.11 Select the next button to perform the energy FWHM and efficiency calibration.
10.6.2.12 Select the Edit Energy button to review the energy and FWHM Calibration.
10.6.2.12.1 Close the Efficiency Calibration side window.
10.6.2.13 Select the save calibration button and save the calibration to Cal\X_Geometry.clb" where X is the detector and geometry is an appropriate geometry name.
10.6.2.14 Enter the calibration description in the format "x_Geometry_Source number_date counted" where X is the detector number and geometry is an appropriate geometry description when prompted. Select the finish button to close the calibration wizard.
10.6.2.15 Print calibration report from the menu "Calibrate\Print Calibration".
10.6.2.16 Select "Analyze", select "Entire spectrum in memory" and file print.
10.6.2.17 Close the spectrum Buffer window and save the spectrum when prompted.

10.6.3 Detector Long Background Counting
10.6.3.1 Remove any samples from the detector, clean the detector, close the shield lid and start acquisition.
10.6.3.2 Select detector in global value quick Start.
10.6.3.3 Select Monthly Background PBC under Automation Groups.
10.6.3.4 Select Background PBC Long Count under Automation Jobs.
10.6.3.5 Login using name and password.
10.6.3.6 Select "OK", ensure detector cave is empty.
10.6.3.7 Repeat for each detector which background you would like to start.
10.6.3.8 After the background is complete it will save as a PBC file.

10.7 Evaluation of Controls for Trends
10.7.1 Identified controls shall be evaluated for trends at a minimum frequency of 1 month, but as often as deemed by analysts. The controls and trending rules are listed below (see appendix for list of trending rules).
10.7.1.1 Background FWHM, Centroid (by engergy), Efficiency
10.7.1.1.1 Trending Rules 2, 3 & 6
10.7.1.2 Source FWHM, Centroid (by engergy), Efficiency
10.7.1.2.1 Trending Rules 2, 3 & 6

11.0 PROCEDURE

11.1 Calibration Quality Control (**Daily Check**)
11.1.1 Place the calibration quality control sample on the detector, and start acquisition.
11.1.2 Select detector from global value quick start.
11.1.3 Select Quality Control under Automation Groups.
11.1.4 Select Daily Quality Control Check under Automation Jobs.
11.1.5 Login with user name and password.
11.1.6 Select "OK", ensure source is on detector.
11.1.7 Repeat for each detector.
11.1.8 Record in the instrument run log.

11.2 Background Quality Control (**Daily Background**)
11.2.1 Remove any samples from the detector, and start acquisition.
11.2.2 Select detector global value quick start.
11.2.3 Select quality control under automation groups.
11.2.4 Select daily background check under automation jobs.
11.2.5 Login with username and password.

11.2.6 Select "OK", ensure detector cave is empty.
11.2.7 Repeat for each detector.
11.2.8 Record in the instrument run log.

11.3 Sample Counting
11.3.1 Place the sample on the detector.
11.3.2 Select detector from global value quick start.
11.3.3 Select analyze samples under automation groups.
11.3.4 Select count sample under automation jobs.
11.3.5 Login with username and password.
11.3.6 Scan sample description from barcode report.
11.3.7 Select analysis method, sample type, geometry, library, correct date, count time, and continue.
11.3.8 Select "OK", ensure sample is on detector.
11.3.9 Record in the instrument run log.

11.4 Analysis/Analyte Specific Considerations
11.4.1 Analysis library
11.4.1.1 The analysis library, if supplied by the laboratory, should use nuclide energy lines and abundances from the table of isotopes edited by Gerhard Erdtmann and Werner Soyka.
11.4.1.2 The analysis library used should be representative of the types of analytes expected to be seen in the project samples.
11.4.1.3 The analysis library may be supplied by the client, defining their project-specific analytes, assumptions, energy lines, and abundances.
11.4.2 Ra-226 analysis using the nuclide's 185.99 keV peak
11.4.2.1 Ra-226 may be analyzed using the 3.28% abundant peak at 185.99 keV. This analysis may be performed using any geometry any time after preparation of the sample (including "0-day"). Ra-226 as a nuclide must be included in the analysis library with the abundance and energy listed.
11.4.2.1.1 Note: The 54% abundant 185.72 keV gamma ray emission of U-235 may produce a significant interference. This can produce high-bias to Ra-226 due to counts from the higher-abundant U-235 peak being mis-classified into the Ra-226 peak. However, there are circumstances where the opposite may occur (Ra-226 counts "lost" into the U-235 peak), producing a low bias. The client should be aware of the potential interferences.
11.4.3 Ra-226 inferred from Bi-214 after 21-days of ingrowth
11.4.3.1 Ra-226 may be inferred from the Bi-214 daughter after ingrowth in a sealed container devoid of headspace. This is the preferred means for determining Ra-226 by gamma spectrometry.
11.4.3.1.1 The ideal ingrowth period is 21-days (or longer), resulting in nearly complete secular equilibrium of the decay chain through Rn-222 down to the Bi-214 progeny. Other client-requested ingrowth periods (e.g. 14-days, 10-days, 7-days) may be used, but a NCM should be written stating the reported Ra-226 result may be low-biased due to the shorter ingrowth period.
11.4.3.1.2 The container should be reasonably well sealed to ensure gaseous Rn-222 does not escape the container.
11.4.3.1.3 The container (geometry) should not have significant headspace, into which Rn-222 could escape, ultimately resulting in a low bias. If such a geometry is used (e.g. due to limited aliquot provided by the client), a NCM should be written.
11.4.4 Other inferences:
11.4.5 Oftentimes nuclides, which may not have any reasonable gamma emissions, are reported based upon either shorter-lived daughters or a longer-lived parent, assuming

secular equilibrium between the two. The half-life used for any decay corrections should be that of the longer-lived parent. Note: Such assumptions should be evaluated by the client as to appropriateness to their sample type. For example, Th-232 is often inferred from the Ra-228 daughter. While this is a good assumption for most soil samples, this is likely not a good assumption for an oil & gas flowback water. Common inferences ("re-maps") include:

input analyte	remapped to analyte
Ac-227	Th-227
Ac-228	Th-232
Ac-228	Ra-228
Ag-108m	Ag-108
Bi-214	Ra-226
Cs-137	Ba-137m
Pb-210	Po-210
Pb-210	Bi-210
Pb-212	Th-228
Pb-212	Ra-224
Rh-106	Ru-106
Sb-125	Te-125m
Th-227	Ra-223
Th-227	Ac-227
Th-227	Bi-211
Th-227	Pb-211
Th-231	U-235
Th-234	Pa-234
Th-234	U-238
U-235	Th-231

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 All calculations are performed in GammaVision-32 software; conversions are performed in RadCapture. Calculations are found in ST-QAM.

12.3 Other Detectable Radionuclides (ODRs - Identified as TICs in LIMS) when requested shall be evaluated when the activity to MDC ratio (Act/MDC) is above two (2) unless specified by the Client Requirement Memo (CRM) or by project notes in LIMS.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (**MB**)

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the requested reporting limit (RL). If no requested limit is assigned to the analyte(s), the activity to MDC ratio (Act/MDC) shall be below two (2). No further action would be required at that point.

13.2.1.2 Project specific requirements, if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the Client Requirement Memo or in project notes in LIMS..

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – If the method blank exhibits activity above the limits/criteria (see Section 13.2.1), the affected analytes shall be evaluated to determine possible force fitted peak, or unsupported energy lines using the Gamma Vision™ raw data reports. If the contaminant is found to be unsupported, the analyst will narrate findings in an NCM.The blank may be re-counted once to confirm the activity. The recount can be used to report only the affected analyte(s); the original count can be used to report all unaffected analyte(s) if the analyst finds no additional error in the original count. If the re-counted MB activity exceeds the criteria/limit further evaluation will be performed and narrated accordingly. Note that certain analytes are common laboratory contaminants which require special narration.

13.3 Laboratory Control Sample (**LCS**)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – The LCS may be re-counted once to confirm the result. If the re-counted LCS exceeds the control limit, samples that are non-detect may be reported with an NCM.

13.3.2.2 LCS Spike Recovery excursion (low) – The LCS may be re-counted once to confirm the result. If the low recovery is confirmed, the batch is recounted.

13.4 Duplicate

13.4.1 Acceptance Criteria:

13.4.1.1 All control analytes must be within the specified control limits for precision (RPD), max. 40% RPD, RER < 1, and/or DER ≤ 3.

13.4.2 Corrective Action for Duplicate not meeting acceptance criteria:

13.4.2.1 RPD/RER Duplicate excursion – The sample is recounted if both RPD and RER (and/or DER) exceed criteria.

13.5 Insufficient Sample

13.5.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. The insufficient sample description is included in the LIMS NCM within the type defining the excursion.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are documented in LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration of capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 28th Edition, method GA-01-R, Gamma Radioassay
17.2 EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method 901.1
17.3 American National Standards Institute (ANSI) Accredited Standards Committee on Radiation Instrumentation, N42; ANSI N42.14-1999, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides
17.4 Ortec MCB Connections-32, Hardware Property Dialogs Manual, current version
17.5 MAESTRO–32, MCA Emulator, current version
17.6 GammaVision–32, Gamma-Ray Spectrum Analysis and MCA Emulator, current version
17.7 Master library Source: Gerhard Erdtmann, Werner Soyka
17.8 TestAmerica Quality Assurance Manual (ST-QAM), current revision
17.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
17.10 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
17.11 Associated SOPs, Current Revision:
17.11.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
17.11.2 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
17.11.3 ST-RC-0025, Preparation of Samples for Gamma Spectroscopy

17.11.4 ST-QA-0002, Standards and Reagent Preparation
17.11.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.11.6 ST-QA-0036, Non-Conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Annual Review, No Changes.
19.2 Revision 8:
- 19.2.1 Increased background count times from 12 to 36 hours in section 10.3.1.1.
- 19.2.2 Updated the procedure for detector long background counting in section 10.5 to reflect new software.
- 19.2.3 Updated daily calibration checks, daily background and sample counting procedures in section 11.0 to reflect new software.

19.3 Revision 9:
- 19.3.1 Replaced quartz sand with sodium sulfate to be used for soil method blanks in section 9.2.
- 19.3.2 Updated section 10.4 regarding instrument daily checks.
- 19.3.3 Updated data assessment and acceptance criteria in section 13.0
- 19.3.4 Updated section 9.0 regarding batch, method blank and laboratory control samples.
- 19.3.5 Updated the calibation points for an internal calibation in section 10.1.
- 19.3.6 Updated the percent recovery regarding the ICV in section 10.2.
- 19.3.7 Updated software storage file name throughout section 10.5.

19.4 Revision 10:
- 19.4.1 Updated references to QuantIMS through out
- 19.4.2 Update §10.1
- 19.4.3 Added §10.3 Annual Calibration Verification
- 19.4.4 Updated §10.4: 36 hour background changed to 12 hour and requirement to complete Attachment 2
- 19.4.5 Added Attachment 2, “Monthly Background Complete” example
- 19.4.6 Updated §13 references to Clouseau changed to LIMS
- 19.4.7 Added §17 reference to ANSI 42.14-1999

19.5 Revision 11:
- 19.5.1 Updated §1.4 with corrected termonolgy
- 19.5.2 Updated §6.0 software details
- 19.5.3 Additon/Update §10.0 major change in calibration
- 19.5.4 Updated §13.0 additional corrective action steps
- 19.5.5 Updated §15.0 with new verbiage

19.6 Revision 12: (04/16/2014)
- 19.6.1 Spelling and grammar corrections made throughout SOP.
- 19.6.2 Sections 10.2.3 and 10.2.5 had wording changed to common text.
- 19.6.3 Section 10.4.3.3 was updated to add ‘for the same specific parameter as the day before’ and ‘until the detector can be evaluated and/or maintenance can be performed.’.
- 19.6.4 Section 10.5.1.2.1 was added to provide limits for monthly backgrounds, which were not previously provided.
- 19.6.5 Section 13.4.2 had ‘LCS’ changed to ‘duplicate’ since it is the duplicate section and LCS was incorrectly referenced.

19.7 Revision 13: (06/22/2015)
- 19.7.1 Section 10.5.1.1 was updated to say “Backgrounds count for a minimum of 12 hours” new wording.

19.7.2 Section 10.6.3.2 updated captialzation “global value quick”.
19.7.3 Section 11.1.2 updated capitalization “global value quick start”.
19.7.4 Section 111.3.2 updated capitalzation “global value”.
19.7.5 Section 11.3.7 updated to say “and continue.” new wording.

19.8 Revision 14: (05/23/2016)
19.8.1 Trending evaulation added to section 10
19.8.2 Appendix with trending rules added

19.9 Revision 15 (5/2/2017)
19.9.1 True Coincidence Correction (TCC) calibration.onformation added to Section 10.1
19.9.2 Trending Rules changed from 2,3,and 5 to 2,3,and 6

19.10 Revision 16 (12/12/2017) Technical Review: R. Mueller/QA Review: M Ward)
19.10.1 Section 10.1.2.55 changed/edited
19.10.2 Section 10.3 removed ACV mention; ACV is no longer performed

19.11 Revision 17 (3/9/2018) Technical Review: T. Romanko/Rachel Mueller / QA Review: M. Ward
19.11.1 Section 10.4.1.1.2 removed no longer utilized
19.11.2 Added Section 11.4: Analysis/Analyte Specific Considerations
19.11.3 Section 9.1 – fixed SOP reference
19.11.4 Added Section 10.1
19.11.5 Section 10.5.1.1.2 removed
19.11.6 Attachment 2 removed (Monthly Background Complete form); no longer posted
19.11.7 Added Section 12.3 – ODR information
19.11.8 Section 13.2 – added Method Blank criteria

19.12 Revision 18 (3/22/2019) Technical Review: R. Mueller/QA Review M. Ward
19.12.1 Section 10.2 – Added verbage regarding calibration frequency
19.12.2 Section 10.2 – reference QSM daily check requirements- two pass criteria.
19.12.3 Section 13.4 – added DER to precision criteria

19.13 Added Eurofins logo and updated copyright information (4/18/2019)

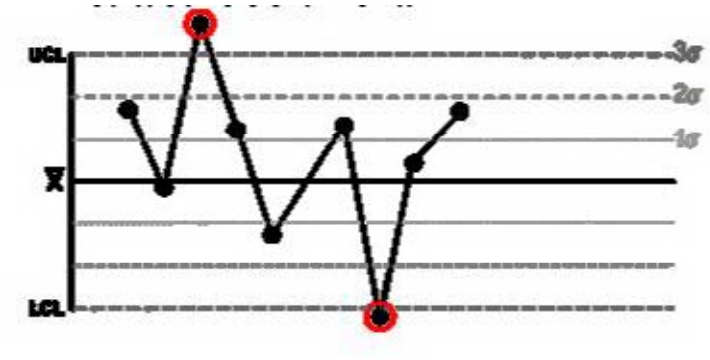
19.14 Revision 19 (11/12/2019) Technical Review – J. Watson ; QA Review – K. Ely
19.14.1 Section 9
19.14.1.1 Updated MB and LCS to match current practice.
19.14.1.2 Removed duplicated paragraph on NCM’s.

Appendix

Rule 1: One point is more than 3 standard deviations from the mean.

~ Simplest: Single point is considered out of control event

- Corrective action required



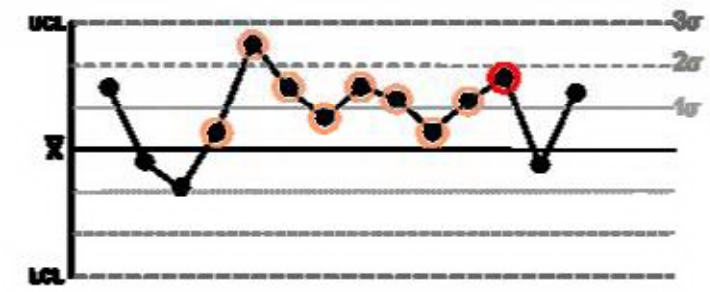
Rule 2: Seven (or defined 'n') points in a row are on the same side of the mean.

~ n = 7 warn trigger

- Mean shift: (same side of mean)

- Some prolonged bias exists.

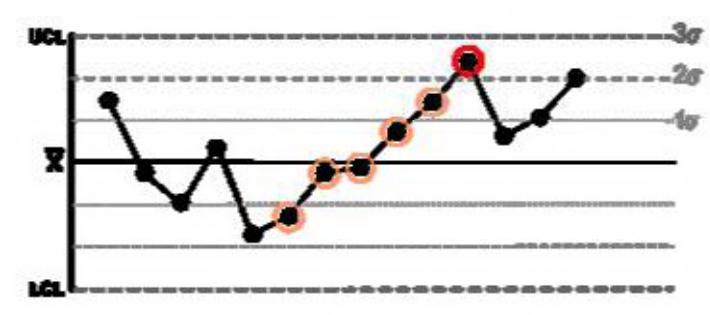
~ Corrective action recommended



Rule 3: Six (or more) points in a row are continually increasing (or decreasing).

- A trend exists.

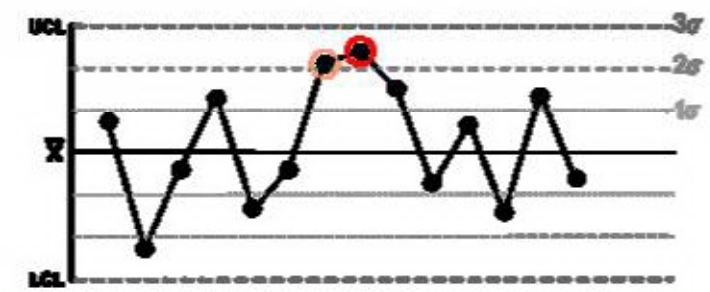
  ~ Corrective action recommended



Rule 5: Three out of three points in a row are more than 2 standard deviations from the mean in the same direction.

- There is a medium tendency for samples to be mediumly out of control.

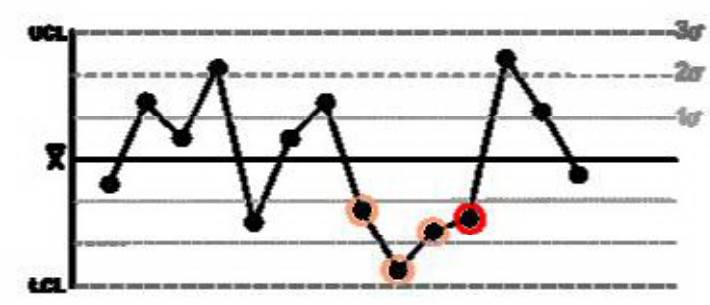
  ~ Investigate for preventive. May not be CAR



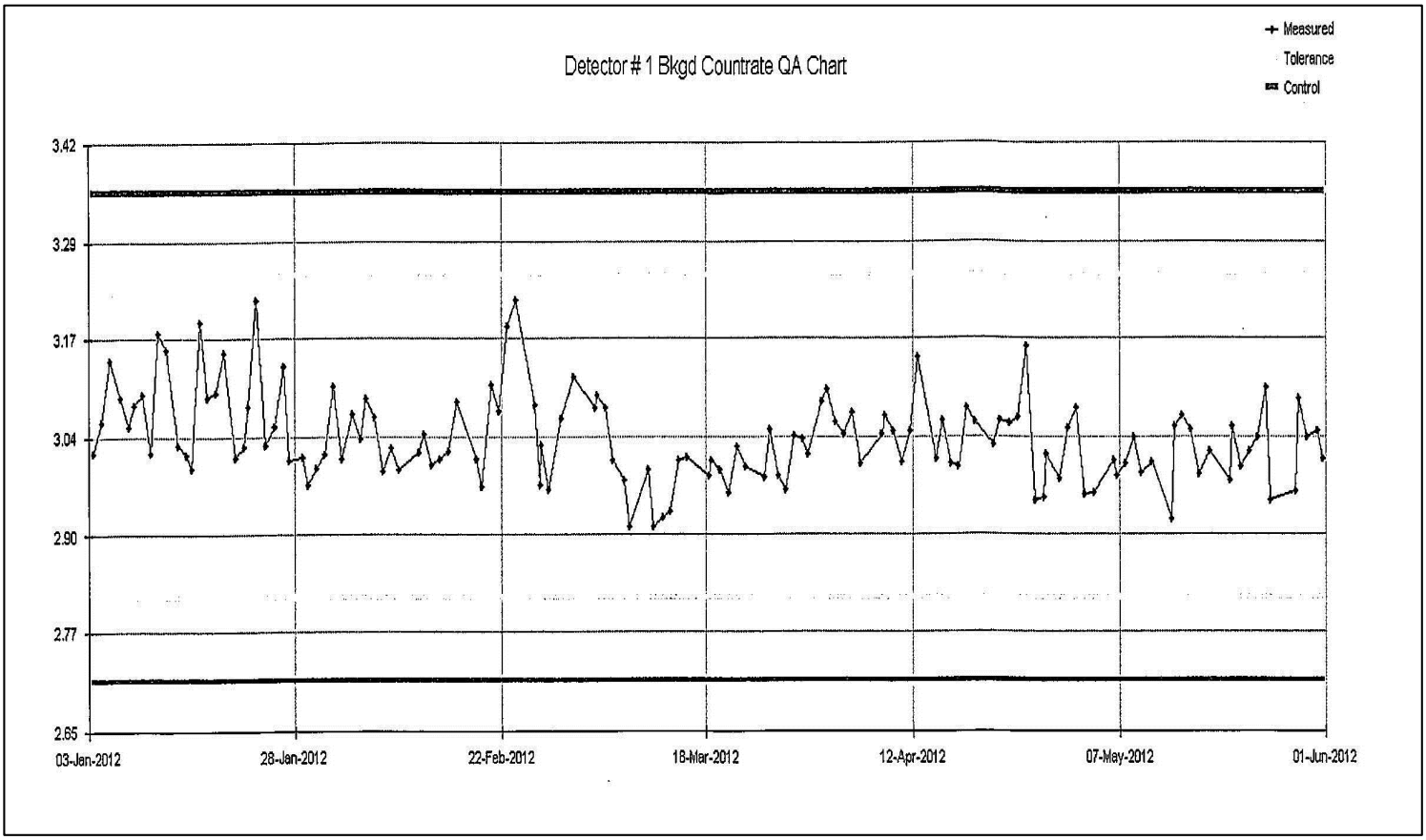
Rule 6: Four out of five points in a row are more than 1 standard deviation from the mean in the same direction.

- There is a strong tendency for samples to be slightly out of control.

  ~ No CAR, Statistical trend



Attachment 1



Title: LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING (GFPC) SYSTEM ANALYSIS

Approvals (Signature/Date):

Jody Watson 11-5-19
Jody Watson Date
Radiochemistry Count Room Manager

Michael Ridenhower 11/6/19
Michael Ridenhower Date
Health & Safety Manager / Coordinator

Kristen Ely 11/6/19
Kristen Ely Date
Quality Assurance Manager

Jodie Carnes 11/6/19
Jodie Carnes Date
Laboratory Manager

This SOP was previously identified as SOP No. ST-RD-0403 Rev. 19

Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to all Low Background Proportional Counting instruments. TestAmerica St. Louis performs radium-226/228, strontium-89/90, gross alpha/beta, and chlorine 36.

1.2 This SOP is based on SW846 method 9310, 9315 and 9320; EPA methods 900.0, 903.0, 904.0, 905.0; Standard Method 7110C and DOE EML HASL 300 method, Ba-01-R, Sr-02 and Sr-03-RC.

1.3 The SOP applies to GFPC analysis of liquid and solid matrices.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This procedure provides instructions for the daily calibration and maintenance of the Low Background Proportional Counting instrumentation.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualifiers.

3.2 IQC - a computerized Quality Control Program where the counting results of Daily Radioactive check sources and Daily Background checks are entered and compared to statistical average data. A measurement within ± 3 standard deviations indicates the detector is operating within acceptable parameters.

3.3 αLL - discriminator setting indicating the alpha lower voltage limit.

3.4 Alpha Voltage Only - detector voltage capable of collecting ions created by alpha radiation only. Ion pairs created by beta radiation are not collected.

3.5 αUL - discriminator setting indicating the instruments alpha upper voltage limit.

3.6 βLL - discriminator setting indicating the beta lower voltage limit.

3.7 βUL - discriminator setting indicating the beta upper voltage limit.

3.8 Crosstalk - a measure of the amount of beta radiation that is collected in the alpha radiation channel; it is also a measure of alpha radiation collected in the beta channel.

3.9 Plateau - a point on a graph of count rate vs. detector bias voltage where further increases in bias will not result in an increase in measured counting rate.

3.10 LB4100 – LBPC (Low background Gas Flow Proportional Counting instrument).

4.0 INTERFERENCES

4.1 A detector contaminated with radioactive material will result in a high background and interfere with the correct measurement of a sample.

4.1.1 If a sample reaches 10,000 counts, and the sample count rate is 60 cpm or greater, then another daily background check is performed on that detector. If the detector background check is unacceptable, the detector is taken Out Of Service until action is taken to bring the background check within acceptable limits.

4.2 The actual counting efficiency for alpha radiation decreases greatly with a density > 6.0 mg/cm2. Therefore, the maximum acceptable mass density is typically 5 mg/cm2 or less that 100 mg for a 2” planchet.

4.3 For beta radiation, reliable data may be obtained counting samples with a density as high as 10 mg/cm2 or greater.
4.4 Sample thickness as well as moisture content may impact the alpha and/or beta results.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Silver Nitrate	Poison Corrosive Oxidizer	$0.01^{g}/_{m3}$ (TWA) for silver, metal dust, and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Skin contact may cause redness, pain, and severe burning. Eye contact can cause blurred vision, redness, and pain.
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH_3)	Inhalation symptoms may include irritation to the respiratory tract. Ingestion symptoms may include pain in the mouth, chest, and abdomen with coughing, vomiting, and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 Low Background Proportional Counter, equivalent to the Canberra/Oxford/Tennelec LB4100, or Protean MPC9604.
6.2 Gas mixture, 90% argon, 10% Methane
6.3 Blank planchets
6.4 PC based data acquisition system, IQC software

6.4.1 See "Maintenance Tracker" database for software version.
6.5 Centrifuge tubes
6.6 Centrifuge
6.7 Vortex
6.8 Pipettes, Eppendorf or equivalent
6.9 Pipette, disposable

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision

7.2 Radioactive sources to measure beta radiation,: Sr-90

7.3 Radioactive sources to measure alpha radiation: Am-241, Am-243, Th-230 and Ra-226

7.4 Deionized Water (DI).

7.5 Silver nitrate ($AgNO_3$), 0.5 N

7.6 Sodium chloride (NaCl), crystals

7.7 Sodium chloride (NaCl), 0.5 N

7.7.1 Add 50 mL of DI water to a 100 mL volumetric, add 5.84 g of NaCl, dilute to 100 mL, cap and shake to dissolve. Adjust volume to 100 mL with DI water.

7.8 Ammonium hydroxide (NH_4OH), concentrated, 28 N

7.9 Ammonium hydroxide (NH_4OH), 5 %

7.9.1 Add 25 mL of concentrated Ammonium Hydroxide to 475 mL of DI water. **CAUTION – Ammonium hydroxide is corrosive. Mist and vapor cause burns to every area of contact.**

7.10 Cl-36: At least four sodium chloride standards are prepared for calibration.
7.10.1 Add 10 mL of DI water to 4 centrifuge tubes.
7.10.2 Add 0.500 mL of 0.5 N sodium chloride carrier solutions to each centrifuge tube. Swirl to mix.
7.10.3 Add 2 drops of 5 % ammonium hydroxide solution, swirl to mix.
7.10.4 Add 12 mL of 0.5 N silver nitrate solution to each centrifuge tube.
7.10.5 Vortex for 30 seconds.
7.10.6 Centrifuge and decant supernate to waste.
7.10.7 Proceed to section 11.4, Planchet Preparation of Silver Chloride Precipitation of SOP ST-RC-0036.
7.10.8 Average the four weights for the sodium chloride carrier solution, record the standardized weight in the log book and on the bottle.
7.10.9 NOTE: It may be necessary to use more than 0.500 mL of carrier in some large water samples or calibrate a 4 N sodium chloride carrier solution. The efficiency of the detectors will have to be calculated using the heavier sodium chloride carrier solution.

7.10.10 Prepare four sodium chloride calibration samples as in Section 7.10 but add a known amount of Cl-36 to each tube before the sodium chloride carrier is added. Analyze samples by GFPC and determine detector efficiency as per Section 12, Data Analysis and Calculations.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
8.2 See associated sample preparation SOPs ST-RC-0020, ST-RC -0021, ST-RC -0036, ST-RC -0040, ST-RC -0041and ST-RC -0050, for more detailed information.

9.0 QUALITY CONTROL

9.1 See GFPC preparation SOPs for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same reagent lots.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank (MB)**

9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 A method blank must be prepared with every sample batch.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 An LCS is a blank matrix spiked with a known amount of target analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 An LCS must be prepared with every sample batch.

9.5 **Matrix Spike**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate**

9.6.1 A Sample Duplicate is an additional aliquot of a field sample, processed simultaneously with, and under the same conditions as, samples through all steps of the analytical

process to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**

9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Additional preventative maintenance can be found in ST-QA-0024.

10.2 Voltage Plateau Determination

10.2.1 **Frequency**:

10.2.1.1 Performed as a part of the Intial Calibration.

10.2.2 **Voltage Plateau Determination on Protean MPC 9604**

10.2.2.1 Place the Sr 90 source or sources in the detector drawer.

10.2.2.2 Select detector of interest on the computer screen.

10.2.2.3 Click Plateau under Count Method.

10.2.2.4 Set time to accumilate at least 10,000 counts for distributed Sr90 source.

10.2.2.5 Select A, B. C, D.

10.2.2.6 Click OK.

10.2.2.7 When count is complete select Plateau under instrument Specific.

10.2.2.8 Set Beta appropriate voltage with arrows </>. Evalulate and Print report.

10.2.3 **Criteria for Plateaus for Protean MPC 9604**

10.2.3.1 Acquire 40 data points in 30V increments beginning at 705V and ending at 1875V. Slope should be no more than 5%.

10.3 **Discriminator Settings**

10.3.1 **Frequency:**

10.3.1.1 Performed as a part of the Intial Calibration.

10.3.2 **Discrimator Settings on Protean MPC 9604**

10.3.2.1 Collect a minimum of 10,000 counts for each of Am-241, Th-230 and/or Po-210 sources

10.3.2.2 Calculate the percentage of crosstalk and compare the results to historical and expected values. Consult the Technical director if the values fall out of range.

10.4 **Initial Calibration (IC)**:

10.4.1 **Frequency**:

10.4.1.1 The Gas Flow Proportional Counter (GFPC) is calibrated prior to initial use, following repair or replacement of a key detector part when subsequent performance checks indicate a change in performance after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in the instrument response, or when indicated by corrective actions.

10.4.2 The specific calibration source preparations can be found in the file containing the previous calibration.
10.4.3 All nuclide sources shall be NIST traceable.
10.4.4 The efficiency calibration shall consist of at least seven mass attenuated calibration standards, unless a single point source efficiency is to be determined.
10.4.5 Alpha, Beta Ra226 - at least seven mass attenuated calibration standards
10.4.5.1 For gross alpha, use Th-230 in the nitrate form (spiked into varying amounts of salt solution, converted to nitrate) .
10.4.5.2 For Ra-226, use Ra-226 standard in sulfate form (co-precipitated with varying amounts of barium sulfate).
10.4.5.3 For gross beta, Sr-90, Total Beta Strontium, and Ra-228, use Sr-90/Y90 in the nitrate form (spiked into varying amounts of salt solution, converted to nitrate).
10.4.5.4 For gross alpha by co-precipitation, use Th-230 standard (spiked into varying amounts of iron [hydroxide] and barium [sulfate] carrier, dissolved into nitric acid, and dried onto the planchet.
10.4.6 Air Filter – single point calibration
10.4.6.1 For alpha use Am-241
10.4.6.2 For beta use Sr-90
10.4.7 Cl-36 – Averaged 4 point calibration.
10.4.7.1 For Cl-36 use Cl-36
10.4.8 The standards shall have enough activity to generate at least 10,000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5,000 counts per second.
10.4.9.1 For alpha and beta analysis, separate sets of calibration sources shall be prepared.
10.4.9 The mass attenuation is accomplished by utilization of a salt solution with comparable make up to the majority of samples seen in the laboratory.
10.4.9.1 Alternatively, the mass attenuation may be accomplished by using the same carrier solution used in a specific analysis.
10.4.10 Each standard shall be counted in every detector to be calibrated.
10.4.11 **Alpha to Beta Crosstalk Determination**
10.4.11.1 The mean mass is determined for each data point used to calculate the mass attenuation curve.
10.4.11.2 These curves are calculated and plotted and the percent of alpha into beta crosstalk is determined. This is done by dividing the beta counts per minute as observed in the beta channel from the alpha calibration source counts by the sum of the alpha and beta counts per minute.
10.4.11.3 The mean percent of alpha into beta is determined for each mass point by using the count data accumulated for two sets of alpha sources.
10.4.11.4 The crosstalk curve is plotted as mean crosstalk values relative to the mean mass for the two sets of data.
10.4.11.4.1.1 In this manner the crosstalk factor can be determined for any given mass.
10.4.11.5 The equation of the curve shall be determined using polynomial functions.
10.4.11.6 The coefficient of determination (R^2) shall be calculated and displayed on the plot as well as the equation for the trendline.

10.4.12 **Beta to Alpha Crosstalk Determination**

10.4.12.1 Since beta to alpha crosstalk does not vary across mass, a mean beta to alpha crosstalk correction factor is calculated.

10.4.12.2 The percent of beta into alpha is determined by dividing the alpha counts per minute as observed in the alpha channel from the beta calibration source counts by the sum of the alpha and beta counts per minute.

10.4.12.3 The mean percent of beta into alpha is determined for all mass points. The mean percent is insignificant in calculating results, therefore is not applied to the result calculation.

10.4.13 **IC Criteria:**

10.4.13.1 The efficiency of the detector (the dependent variable) shall be plotted on a single graph against the masses (the independent variable) for all data points.

10.4.13.2 The equation of the calibration curve shall be determined using polynomial functions and be included on the plot of the curve. The curve shall be continuous and smooth.

10.4.13.3 The degree of the polynomial shall not exceed three. The number of discreet source pairs shall be two more than the degree of the polynomial.

10.4.13.4 The percent difference of the measured efficiency and theoretical efficiency shall be calculated for all data points.

10.4.13.5 Points that are visual outliers or demonstrate greater than 15 percent difference between the measured efficiency and theoretical efficiency may be removed at the analyst's discretion. Low residual mass sources and samples are difficult to plate with acceptable duplicate precision. Therefore, high outliers may not necessarily be removed from the calibration if they mimic live sample masses. In any case outliers above 15 percent shall be removed from the calibration curve. No more than 20 percent of the data points may be removed. Reasons for removal or inclusion of outliers shall be documented in the calibration narrative. Once outliers are removed, the percent difference between the measured efficiency and theoretical efficiency must be recalculated using the new polynomial coefficients generated from removal of data points. If outliers over 15 percent difference remain between the measured efficiency and theoretical efficiency the Radiochemistry Manager/QA must be consulted before calibration may continue.

10.4.13.6 The coefficient of determination (r^2) shall be calculated and displayed on the plot with the equation of the trend line. An r^2 greater than or equal to 0.9 is required to proceed to counting of verification sources.

10.5 **Independent Calibration Verification (ICV)**

10.5.1 **Frequency**:

10.5.1.1 Performed with every intial calibration

10.5.2 GFPC initial calibrations must be verified by a second source standard.

10.5.3 The ICV standard is NIST traceable.

10.5.4 The ICV is counted to accumulate at least 5,000 counts.

10.5.5 **ICV Criteria**:

10.5.5.1 Prepare 3 verification sources varying in expected mass (low, medium and high) within the calibration range of the curve, unless a single point source is to be determined.
10.5.5.2 Alpha and Beta 3 sources
10.5.5.3 Ra226 Minimum of one source
10.5.5.4 Air Filter Minimum of one source
10.5.5.5 Cl-36 – Minimum of one source
10.5.5.6 The source/standard used for the ICV shall be from an independent second source as defined within the laboratory Quality Assurance Manual."
10.5.9.7.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.
10.5.5.7 Count the secondary source in all detectors that were calibrated.
10.5.5.8 Calculate the results in terms of percentage recovery.
10.5.5.9 Calculate the mean results of all masses across each detector.
10.5.5.10 **Criteria:**
10.5.9.11.1 Individual points are within 30 percent of the true value
10.5.9.11.2 The mean result of all masses across all detectors is less than 10 percent.
10.5.9.11.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.6 **Setting Performance Check Criteria After Calibration**
10.6.1 Twenty background check samples are counted and used to establish quality control limits for the daily background checks.
10.6.2 The limits for the background check sample will be established with five points from four months. Every month the oldest months points will be removed and points from the current month will be added.
10.6.3 Twenty alpha/beta check sources are counted after calibration and used to establish quality control limits for the daily source checks.
10.6.4 The limits for alpha/beta check sources will be a running average of the four months post calibration.
10.6.4.1 The limits are to be documented.
10.6.4.2 The limits will be re-established monthly at the following frequency
10.6.4.2.1.1 1st month – take first five data points from the new month and fifteen data points from the initial calibration.
10.6.4.2.1.2 2nd month – take first five points from new month, five from prior month and ten from initial calibration.
10.6.4.2.1.3 3rd month – take first five points from new month, five points each from the previous two months and five from the initial calibration.
10.6.4.2.1.4 4th month – take first five data points from new month and five points each from the previous three months.
10.6.4.3 Limits are set.

10.7 **Long Monthly Background (ICB)**
10.7.1 **Frequency**:
10.7.1.1 Monthly or whenever instrument conditions have significantly changed since the previous background was performed (e.g. detector replaced, etc.)
10.7.1.2 Minimum count time: 1000 minutes.

10.7.2 Wash the planchet holder and clean the drawers with a 20% radiac wash or ethyl alcohol.

10.7.2.1 Do not spray cleaner directly onto the drawers. Spray cleaner on a Kimwipe, a cotton ball, or paper towel and wipe out the drawers.

10.7.3 Check that instrument settings are as specified in 11.1.

10.7.4 **Orange, Protean, Purple, Red and Blue Long Background Count Set-Up**

10.7.4.1 Select detector 0
10.7.4.2 Select 'sources'→ Start
10.7.4.3 Select 'ICB' by clicking on the Type List arrow.
10.7.4.4 Ensure count time is set to 1000 minutes.
10.7.4.5 Select 'start'
10.7.4.6 Continue these steps with detectors 1-23.
10.7.4.7 Review the data for acceptance when the backgrounds are complete.

10.7.5 **Printing Orange, Protean, Red, Purple and Blue Long Backgrounds**

10.7.5.1 Select 'Data
10.7.5.2 Select 'Source Count Data'
10.7.5.3 Select QC Chart →ICB; Monthly Background
10.7.5.4 Select 'This Range' and enter the date range that the Long Backgrounds (ICBs) were performed and select Refresh
10.7.5.5 Select 'Refresh'
10.7.5.6 Select 'Source Count Summary' under Reports
10.7.5.7 Select source count detail from the Report menu and 'Print'
10.7.5.8 Export to TALs using the Rad Instrument Exporter by editing the file name with _ICB (ex. 10102018_ICB)

10.7.6 **Long Background Criteria:**

10.7.6.1 Long backgrounds are evaluated at ± 3 sigma.
10.7.6.2 The data report is evaluated per detector.
10.7.6.3 If a detector is above this limit, discard planchet.
10.7.6.4 Clean the planchet holder with radiac wash, ethyl alcohol or a detergent spray cleaner and dry thoroughly.
10.7.6.5 Place a clean planchet in the holder and repeat steps for that detector (s) only.
10.7.6.6 Perform a new background.
Note: The detector is tagged with an out of service tag noted with ICB and date. Detector is out of service until a successful background has been achieved.

10.8 Evaluation of Controls for Trends

10.8.1 Identified controls shall be evaluated for trends at a minimum frequency of 1 month, but as often as deemed by analysts. The controls and trending rules are listed below (see appendix for list of trending rules)

10.8.1.1 Alpha background counts – Trending Rules 2, 3 & 6
10.8.1.2 Alpha source counts – Trending Rules 2, 3 & 6
10.8.1.3 Beta background counts – Trending Rules 2, 3 & 6
10.8.1.4 Beta source counts – Trending Rules 2, 3 & 6

11.0 PROCEDURES

11.1 Initial Setup

11.1.1 Check the normal instrument settings for all controls as described below:
11.1.1.1 Tank Flow 8 psi
11.1.1.2 Flow Cells >/= 0.3 SCFH, the flow will vary, the target range is 0.15 to 0.20 SCFH.
11.1.2 The High Voltage is set as indicated in the Manuals for the LB4000/LB4100 located in the count room file cabinet. The Gas Flow Proportional counting system remains as set by the manufacturer and does not require adjustment.
11.1.3 If counting gas has just been turned on, allow a minimum purge time of 30 minutes prior to operation. Record gas tank changes in P-10 tank log book.

11.2 Record date of Daily Background and Check Source Data in Maintenance Tracker found in the QA Public Folder..

11.3 Maintenance
11.3.1 Change out the counting gas when the gauge reads under 500 psi. . Record gas tank changes in P-10 tank log book.
11.3.2 Allow gas to purge a minimum of 30 minutes prior to operation.
11.3.3 Background and checksource checks are required following a gas bottle change.

11.4 Data Acquisition: Daily Background Check and Source Check

11.4.1 **Daily Background Check**: **Orange, Protean, Red, Purple and Blue Instruments**

11.4.1.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.
11.4.1.2 Select detector 0.
11.4.1.3 Select 'sources ' → Start
11.4.1.4 Select or scan 'CCB' by clicking on the Type List arrows.
11.4.1.5 Select 'CCB' by clicking on the file list arrows for purple.
11.4.1.6 Select 'start'
11.4.1.7 Repeat these steps with detectors 1-23.

11.4.2 **Daily Background Criteria:**
11.4.2.1 Review the IQC report for each detector.
11.4.2.1.1 If a detector fails background criteria (3 sigma), clean the detector with radiac wash or ethyl alcohol and re-count.
11.4.2.1.2 Tag detector out of service with an OOS tag noted with "Bkg RC".
11.4.2.1.3 If detector fails Background re-count tag detector with an out of service tag noted with date to indicate that the detector is out of service for the day.

11.4.3 **Daily Source Check Orange, Protean, Red, Purple and Blue Instrument**:

11.4.3.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15. Slowly insert each drawer into the instrument.
11.4.3.2 Select detector.
11.4.3.3 Select 'sources' → Start

11.4.3.4 Scan the corresponding alpha or beta source ID into the Type List. Use the alpha barcode list for starting alpha souces and the beta barcode list for starting beta sources.

11.4.3.5 Select 'start'

11.4.3.6 Repeat these steps for detectors 0-7 for alpha and 8-15 for beta using the correlating detector number and the corresponding source ID..

11.4.3.7 Slowly open each detector drawer when counting is complete. Place beta sources in detectors 0-7 and place alpha sources in detectors 8-15.

11.4.3.8 . Repeat steps 11.5.1.2 to 11.5.1.6 with beta sources in detectors 0-7 and alpha souces in detectors 8-15.

11.4.3.9 Repeat steps 11.5.4.1 to 11.5.4.8 for detectors 16-23.

11.4.3.10 Remove sources from detector drawers when counting is complete.

11.4.3.11 Review the IQC report for each detecctor.

11.4.3.12 Limits +-3% (fail)

11.4.3.12.1 Each analyst loading samples will verify that detectors are in service by reviewing the report in IQC for that day.

11.4.4 **Daily Source Criteria:**

11.4.4.1 Review and save with your name and date on the IQC report for each detector.

11.4.4.1.1 If a detector fails criteria, re-count source.

11.4.4.1.2 If detector fails source re-count tag detector with a Red out of service tag noted with date to indicate that the detector is out of service for the day

11.4.5 **Daily check Criteria:**

11.4.5.1 Review and save with your name and date on the IQC report.

11.4.5.1.1 The indiviuals loading samples will verify that detectors are in-service prior to loading on them.

11.4.5.1.2 In addition Daily checks will be verified at 1st level review of Data.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 Result calculations are performed by TestAmerica St. Louis' Rad Capture software program. These calculations are found in the TestAmerica St. Louis ST-QAM.

12.3 To calculate the efficiency of the detectors for Cl-36, divide the net counts determined of the spiked Sodium Chloride, by the known dpm of the Standard used.

$$\frac{Net\ Counts\ of\ Spiked\ Silver\ Chloride}{Known\ dpm\ of\ Cl-36\ (decay\ corrected\ to\ day\ counted)} = Efficiency$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – (e.g. reprep/reanalysis, narration). If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL (or MDC when applicable) or greater than five times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed..

13.3.2.2 LCS Spike Recovery excursion (low) the batch is re-prepped and re-analyzed for the affected isotope.

13.4 RPD/RER Duplicate excursion – For the RPD/RER (or DER when requested by the client),one or both must be with in acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits (the DER is 3 or less depending on the significant digits). Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (i.e. nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narated in an NCM.

13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.5.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample Result Evaluation

13.6.1 Carrier recovery must be within specified limits.

13.6.2 Carrier recovery low– Samples must be reextracted. Exceptions can be made and results reported with approval from the technical director, manager, or client and appropriate NCM included.

13.6.3 Carrier recovery high

13.6.3.1 A sample carrier recovery outside QC limits may be accepted if the sample results are determined valid by technical director, manager, and/or client approval:

13.6.4 If the sample carrier recovery is significantly higher than normal, the native concentration in the sample of the carrier analyte may be present causing a high bias to the carrier recovery. This high bias to the carrier analyte would in turn cause a low bias to the samples result. The laboratory defines significant to be an additional 20% above

the average LCS/MB carrier recovery (as determined from a population of LCS and MB data), with a maximum of 110%. The table below shows the limits determined for each carrier analyte. The analyst should ensure that the carrier analysis is requested to determine native concentration for samples exceeding the limit.

Radium	Strontium	Chloride
110%	107%	109%

13.6.5 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.7 Insufficient Sample

13.7.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 ANSI N42.25-1997 – American National Standard Calibration and Usage of Alpha/Beta Proportional Counters

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Ba-01-R, Beta Radioassay, Sr-02 Strontium 90, Sr-03-RC Strontium-90 in Environmental Samples.

17.3 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 1, Method 900.0 Gross Alpha and Gross Beta Radiochemistry

17.4 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0 Alpha-Emitting Radium Isotopes

17.5 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0 Radium-228

17.6 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water

17.7 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9310, Gross Alpha and Gross Beta

17.8 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9315, Alpha-Emitting Radium Isotopes

17.9 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9320, Radium-228

17.10 Standard Method 7110C Coprecipitation Method for Gross Alpha Activity in Drinking Water, 19th Edition

17.11 TestAmerica St. Louis Quality Assurance Manual, current revision

17.12 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions

17.13 Associated SOPs, current revisions:

- 17.13.1 ST-PM-0002 “Sample Receipt and Chain of Custody”
- 17.13.2 ST-QA-0002, “Standards and Reagent Preparation.”
- 17.13.3 ST-QA-0024, “Preventative Maintenance”
- 17.13.4 ST-QA-0036, “Non-Conformance Memorandum (NCM) Process”
- 17.13.5 ST-RC-0004, “Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis”.
- 17.13.6 ST-RC-0020, “Determination of Gross Alpha/Beta Activity”
- 17.13.7 ST-RC-0021, “Gross Alpha Radition in Water using Copreciptation”
- 17.13.8 ST-RC-0036, “Determination of Chlorine-36 in Various Matrices by GFPC”
- 17.13.9 ST-RC-0040, ‘Total Alpha Emitting Isotopes of Radium”
- 17.13.10 ST-RC-0041, “Radium-226 and Radium-228 by Chemical Separation Preparation”
- 17.13.11 ST-RC-0050, “Aqueous and Soil Sample Preparation for Strontium-89, Strontium-90, and Total Strontium”

18.0 MODIFICATIONS TO THE REFERENCE METHOD

18.1 Strontium-89 short half life makes it impractical to use as a calibration standard for both radium-228 analysis, as stated in EPA method 904 and SW method 9310, and strontium-89 analysis, as stated in EPA method 905. TestAmerica St. Louis uses a mixed strontium-90/yittrium-90 standard

for its' GFPC beta calibration used in Gross Beta, strontium-90, strontium-89, and radium-228 analyses. TestAmerica St. Louis has selected the strontium-90/yittrium-90 standard because it produces a stable beta emission which can be reliably used for initial and continuing calibration. By using this standard mix, we have beta emissions at the lower and upper energetic spectrum whose average is in the middle of the beta range.

18.2 For Ra-228 analysis, TestAmerica St. Louis uses chemical separation techniques to eliminate other potential beta emitters.

18.3 TestAmerica St. Louis does not perform a direct strontium-89 analysis. TestAmerica St. Louis provides calculated results based on the difference between Total strontium and strontium-90.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated Section 10 to address voltage increase per step, plateau slope and QC check count requirements (5000 counts)

19.2 Rev. 11;
- 19.2.1 Added instument Purple throughout section 10 and 11.
- 19.2.2 Adjusted procedure steps throughout section 11.

19.3 Rev. 12,
- 19.3.1 Added Sr-02-RC and Sr-03-RC to sections 1.0 and 17.0.

19.4 Rev. 13:
- 19.4.1 Added Neptunium to scope in section 1.0.
- 19.4.2 Updated the Quality Control Program for counting daily rad checks and daily background checks in section 3.0.
- 19.4.3 Updated background count set-up, printing and entering protean data in section 10.8.

19.5 Rev. 14: (9/12/2013)
- 19.5.1 Removed references to Clouseau, SAC and QuantIMS
- 19.5.2 Section 5.0 added silver nitrate and ammonium hydroxide
- 19.5.3 Section 6.0 updated to include additional equipment
- 19.5.4 Section 7.0 updated to include addition reagents
- 19.5.5 Section 9.0 added reference to prep SOPs for additional information
- 19.5.6 Section 10.0 added sodium cloride standard preparation & reference to ST-QA-0024
- 19.5.7 Section 12.0 added Cl-36 detector efficiency calculation
- 19.5.8 Section 13.0 updated to include actual corrective actions and native concentration carrier requirements
- 19.5.9 Section 13.0 updated to include corrective actions
- 19.5.10 Section 17.0 added reference to ST-QA-0024

19.6 Rev. 15: (1/16/2015)
- 19.6.1 Added Section 7.10
- 19.6.2 Updated Section 9.6.1
- 19.6.3 Updated Section 10
- 19.6.4 Updated Section 11
- 19.6.5 Added ANSI N42.25-1997 reference to section 17

19.7 Rev. 16: (05/05/2015)
- 19.7.1 Section 11.3.3 added Background and checks are needed following a tank change.

19.8 Rev. 17: (05/23/2016)
- 19.8.1 Section 10, addition of trending of controls
- 19.8.2 Appendix created with trending rules

19.9 Revision 18 05/02/2017

19.9.1 Section 10.2 changes timefrom 5 minutes to “accumilate at least 10,000 counts” for distributed Sr90 source
19.9.2 Section 10.5 changed “single source” to “minimum of one source” for ICV/ACV
19.9.3 Section 10.9 – Added “Protean” source check instructions to those for “Orange” and “Purple” to combine all instruments. Added instrument “Blue” as well.
19.9.4 Section 11.3: added requirement to record gas tank changes on document on separate sheet
19.9.5 Updated SOPs in Section 17

19.10 Revision 19: (12/15/2017 (Tech Review: R. Mueller/J. Watson; QA Review: M. Ward))
19.10.1 Section 5: changed MSDS to SDS
19.10.2 Section 7:
19.10.2.1 Removed Ra-228 from beta calibration standard
19.10.2.2 Removed reference to Milli-Q system
19.10.3 Section 10.5.5 was removed to eliminate the need for ACV
19.10.4 Section 10.5.9 was revised to remove ACV
19.10.5 Section 18.0 removed- the lab changed the source for Ra-226 calibrations from Th-230 to Ra-226 and no note is needed to explain the use of a different analyte for calibration.

19.11 Revision 20: (1/16/2019)
19.11.1 Technical Review: Rachel Mueller/QA Review M Ward
19.11.2 Removed Neptunium calibrations throughout SOP- this is no longer determined by GFPC.
19.11.3 Fixed grammar, miss-types, and formatting throughout SOP.
19.11.4 Re-worded language throughout SOP to be more clear
19.11.5 Section 10.4: changed the language to describe the frequency of initial calibrations.
19.11.6 Section 10.4: moved cross talk section (sections 10.7 and 10.8) to the initial calibration subsection as this is determined during intial calibrations.
19.11.7 Section 10.4.5.4- added Co-precipitation calibration
19.11.8 Section 132.2.1: method blank criteria changed in accordance with QSM (5X)
19.11.9 Section 13.3.1- precision removed- mentioed in section 13.4
19.11.10 Section 13.4- added DER
19.11.11 Section 13.6: removed mention of tracer recovery and criteria- there are no current methods that use tracer recoveries for GFPC, the criteria mentioned is only meant for methods with tracers
19.11.12 Added reference to Standard Method 7110C to Sections 1 and 17

19.12 Added Eurofins logo and udpated copyright information (4/18/2019)

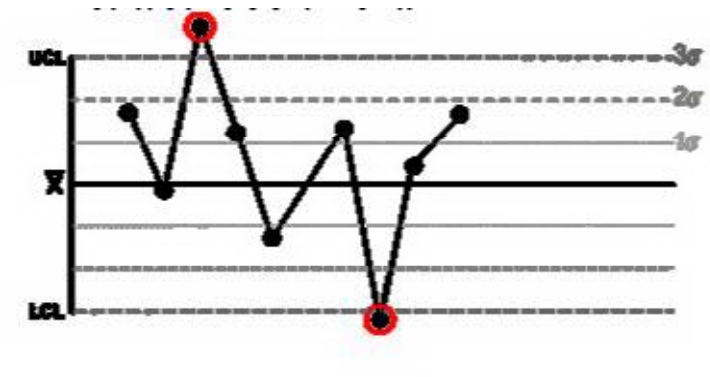
19.13 Annual Review (11/7/2019) Tech Review Jody Watson/QA Review Marti Ward
19.13.1 No Technical changes
19.13.2 Added new GFPC Instrument “Red” to Sections 10 and 11

Appendix

Rule 1: One point is more than 3 standard deviations from the mean.

~ Simplest: Single point is considered out of control event

- Corrective action required



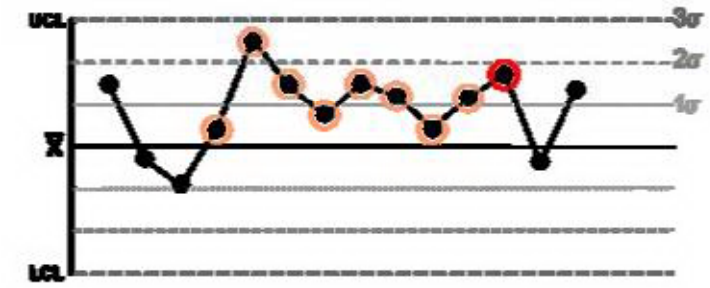
Rule 2: Seven (or defined 'n') points in a row are on the same side of the mean.

~ n = 7 warn trigger

- Mean shift: (same side of mean)

- Some prolonged bias exists.

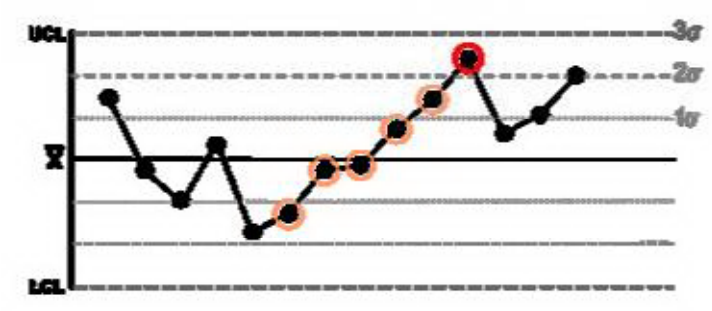
~ Corrective action recommended



Rule 3: Six (or more) points in a row are continually increasing (or decreasing).

- A trend exists.

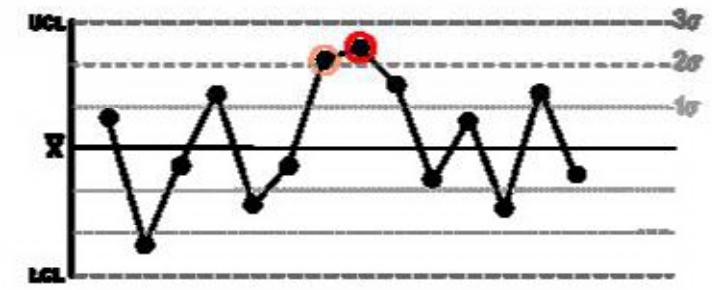
  ~ Corrective action recommended



Rule 5: Three out of three points in a row are more than 2 standard deviations from the mean in the same direction.

- There is a medium tendency for samples to be mediumly out of control.

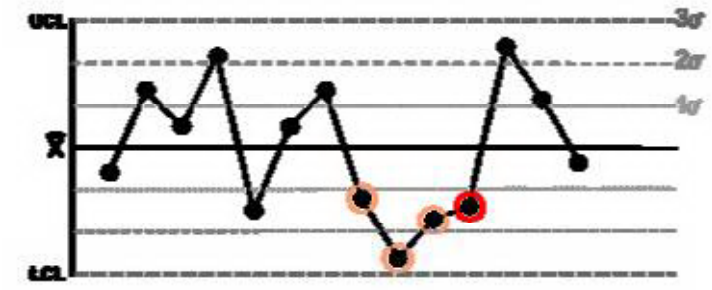
  ~ Investigate for preventive. May not be CAR



Rule 6: Four out of five points in a row are more than 1 standard deviation from the mean in the same direction.

- There is a strong tendency for samples to be slightly out of control.

  ~ No CAR, Statistical trend



eurofins	Always check on-line for validity. **Soxhlet Extraction Procedure for HRMS Analysis in a Solid matrix**	Level: **Work Instruction**
Document number: **T-HRMS-WI9488**		
Old Reference: **1-P-QM-WI-9038122**		
Version: **1**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **22-JUN-2016**	Document users: **5_EUUSLA_HRMS_Manager, 6_EUUSLA_HRMS_Analyst, 6_EUUSLA_HRMS_Data_Reviewers, 6_EUUSLA_HRMS_Sample_Prep**	Responsible: **5_EUUSLA_Specialty Services_Manager**

LIMS ID
Analysis 11030, 13234

US_6_EUUSLA_HRMS_Analyst (Org, 6_EUUSLA_HRMS_Analyst)
Joel Denlinger
Joseph Anderson
Michael Ziegler

Revision Log

Revision: 01		**Effective Date:**	This version
Section	**Justification**		**Changes**
			New

Reference

1. EPA Method 1613B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, October 1994.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 8290A, Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS), February 2007 Rev 1.

3. EPA Method 1668C, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, April 2010

4. EPA Method 1668 Revision A, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, August 2007

5. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title

eurofins	Always check on-line for validity. **Separatory Funnel Extraction Procedure for HRMS Analysis in an Aqueous Matrix**	Level: **Work Instruction**
Document number: **T-HRMS-WI12032**		
Old Reference: **1-P-QM-WI-9038109**		
Version: **2**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **24-OCT-2016**	Document users: **5_EUUSLA_HRMS_Manager, 6_EUUSLA_HRMS_Analyst, 6_EUUSLA_HRMS_Data_Reviewers, 6_EUUSLA_HRMS_Sample_Prep**	Responsible: **5_EUUSLA_Specialty Services_Manager**

LIMS ID
Analysis 10914, 13235

Revision Log

Revision: 2		**Effective Date:** This version
Section	**Justification**	**Changes**
Throughout Document	Reflects current scans	Added scan 13234
Reference	Reflects current references	Added reference to the USEPA Drinking Water Manual
Sample Collection, Preservation, and Handling	Reflects current practice	Added text indicating that drinking water extracts must be analyzed within 40 days of extraction
Procedure B.2.e.	Response to Agency audit	Added a note to the procedure outlining preparation and documentation of the percent solids reference standard

Revision: 01		**Effective Date:** Jun 28, 2016
Section	**Justification**	**Changes**
		New

eurofins	Always check on-line for validity. **Determination of Tetra- Through Octa-Chlorinated Dioxins and Furans using HRGC/HRMS by EPA 1613B or SW-846 Method 8290A**	Level: **Work Instruction**
Document number: **T-HRMS-WI9476**		
Old Reference: **1-P-QM-WI-9015119**		
Version: **10**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **24-AUG-2017**	Document users: **6_EUUSLA_HRMS_Analyst, 6_EUUSLA_HRMS_Data_Reviewers**	Responsible: **5_EUUSLA_HRMS_Manager**

Revision Log

Revision: 10		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version.
Throughout Document	Reflects current document numbers	Deleted old document numbers and replaced with D4 document numbers
References	Updated method	Added 8000D

eurofins	Always check on-line for validity **Ultrasonic Extraction of Chlorinated Herbicides by Method 3550B/C in a Solid Matrix**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10912**		
Old Reference: **1-P-QM-WI -9013472**		
Version: **11**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **11-AUG-2016**	Document users: **5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Herbicide Waters and , 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_Herbicide Chem**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID
Analysis 4181

Revision Log

Revision: 11		**Effective Date:** This version
Section	**Justification**	**Changes**
Document Title	Added method to title	Revised document title to *Ultrasonic Extraction of Chlorinated Herbicides by Method 3550B/C in a Solid Matrix*
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects current scans	Removed analysis #5592 due to deactivation
Definitions	Common terms defined in higher- level documents	Removed definitions and defined first use of acronyms in document

Revision: 11	**Effective Date:** This version	
Sample Collection, Preservation, and Handling	Reflects current sample storage conditions	Revised temperature for storage of samples
Reagents and Standards	Reflects current reagent storage conditions and shelf life	Added information regarding storage conditions and shelf life for reagents
Procedure 2.	Reflects current process	Added information regarding obtaining a sample aliquot
Procedure 13.	Reflects current process	Added language to describe rate of concentration
Procedure 25.b.(11)	Reflects current extract storage conditions	Revised temperature of storage of extracts

Revision: 10	**Effective Date:** Jul 29, 2014	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P -QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Procedure 4.b.	Reflects current procedure	Corrected spiking requirements.
Procedure 25.b (11)	Clarification	Added hold time for extracts.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Methods 3550C, February 2007.
2. Test Methods for Evaluating Solid Wastes, SW-846 Methods 3550B, December 1996.
3. *Sonicator Ultrasonic Processor and Cell Disruptor Operations Manual*, Sound Heat Systems, Inc., 1985.
4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #2487	Food and Tissue Preparation
Analysis #10401	Chlorinated Herbicides by 8151A in Solids by GC-ECD
1-P-QM-PRO-9015405	Ultrasonic Processor Maintenance and Tuning
1-P-QM-PRO-9015475	Glassware Cleaning for Organic Extractions
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Scope
This method is used for the extraction of chlorinated herbicides in soils and solid wastes.

Basic Principles
A portion of sample is placed in a beaker. Acidified sodium sulfate is added to absorb any water present. The mixture is acidified with hydrochloric acid. Surrogate standards are added to each sample to monitor recovery. An aliquot of solvent is then added to the sample. The sample is subjected to sonic disruption to disperse the soil and force solvent contact. The organic compounds present in the soil dissolve in the solvent that is then removed. The sample is extracted two additional times with fresh solvent. The solvent fractions are filtered into a flask containing acidified sodium sulfate. The filtrate is then poured through a funnel containing glass wool to remove the sodium sulfate and into a Kuderna-Danish (K-D) assembly. The sample is concentrated on a steam bath, then 37% KOH and reagent water are added and the sample is again concentrated to remove all solvent.

The aqueous solution is transferred to a separatory funnel and extracted with methylene chloride. The solvent is discarded and the aqueous solution is acidified with sulfuric acid, and extracted with ethyl ether. The ether fractions are placed in a flask containing acidified sodium sulfate and left to sit for at least 2 hours. Then the solvent is transferred to a K-D apparatus and concentrated to 10 mL.

Methanol is added and the sample is subjected to esterification. The extract is then concentrated to 2 mL using nitrogen blow down technique. Hexane is added to adjust the final volume and the extract is florisiled.

Reference Modifications
1. EPA Method Deviation from section 7.2.1.5: (Prior to Procedure 12) The 2-hour wait at this point is not necessary since samples always go through the hydrolysis step. The sample is not centrifuged since the extract is vacuum filtered through filter paper. This eliminates fine particles in the extract.

2. Fisher G6 filter paper is used in place of Whatman #1 filter paper. Fisher G6 is a glass fiber filter paper that is baked at 400°C. This ensures inertness of filter paper.

3. This extraction is sufficient to force the herbicide salts into the aqueous solution. Additional extraction with methylene chloride has been found to be unnecessary and results in a loss of hexachlorophene.

4. Room temperature sulfuric acid is added in place of cold sulfuric acid. There has been no benefit found using cold sulfuric acid in place of room temperature sulfuric acid.

5. Refluxing the samples for 30 minutes at 80°- 85°C has been found to be sufficient to drive off the solvent while providing acceptable recoveries in Procedure 15.

6. Isooctane is not added since the final volume is 10 mL, not 4 mL as written in the method. The isooctane is added to prevent solvent from "blowing off" during methylation. However, since the final volume is greater than the EPA method volume, the isooctane is not needed.

Interferences

Method interferences are caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Always add acid to water to reduce fuming and bumping. The 37% KOH gets hot when prepared. Always wear gloves when handling this solution. In order to reduce the heating of the solutions, the deionized water is chilled in an ice bath prior to preparation of these reagents.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Always wear gloves when handling the diazald and avoid inhaling diazomethane gas. Both are extremely toxic, severely irritating, and have been cited as carcinogens. See specific safety instructions for this procedure listed in the esterification section.

Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are then transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers. All waste generated from esterification must be placed in a beaker in the hood and only be added to the solvent waste stream in the lab-wide disposal facility.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass jars with PTFE-lined lids and stored refrigerated at 0 - 6°C, not frozen prior to extraction. Samples must be extracted within 14 days of collection.

Apparatus and Equipment

1. Sonic Probe apparatus (with a minimum of 300W output) for extracting organic components from a soil matrix

2. Kuderna-Danish (K-D) assembly with appropriate ampule for concentrating the solvent used during concentration

3. Steam bath – VWR/LLI Model #1127 or equivalent

4. Filter paper – Fisher G6 or equivalent. The filter paper is baked at 400°C for 4 hours.

5. N-Evap with nitrogen supply

6. 125-mL separatory funnel

7. pH meter or paper – assorted ranges

8. Diazomethane generator

9. Boiling chips, Teflon® and glass beads

10. Vials – assorted sizes

11. Beakers – Stainless steel, assorted sizes

12. Pipettes – Class A, assorted sizes

13. Graduated cylinders, Class A, assorted sizes

14. Solvent dispenser – Brinkmann, adjustable

15. Pipettes – disposable

16. Balance – capable of weighing to 0.01 g

17. Wash bottles – Teflon®

18. Volumetric flasks – Class A, assorted sizes

19. Erlenmeyer flasks – assorted sizes

20. Syringes – assorted sizes

21. Micropipetter

22. Test Tubes

Reagents and Standards

1. Methylene Chloride – pesticide grade or equivalent. Store at room temperature for up to one year.

2. Acetone – pesticide grade or equivalent. Store at room temperature for up to one year.

3. Hexane – pesticide grade or equivalent. Store at room temperature for up to one year.

4. Ethyl ether, high purity, nonpreserved – Store at room temperature. Follow manufacturer's expiration information.

5. 25% Sulfuric Acid – pesticide grade or equivalent. Store at room temperature for up to one year.

6. Hydrochloric Acid – ACS grade. Store at room temperature for up to one year.

7. Diazald (N-methyl-N-nitroso-p-toluenesulfonamide)-Store 0 - 6°C, not frozen for up to 5 years.

8. Reagent Alcohol GR- ACS grade. Store at room temperature for up to 1 year.

9. Potassium hydroxide, (KOH) (37% w/v)

   a. Dissolve 37 g of ACS grade KOH into approximately 80 mL of reagent water in a 100-mL volumetric flask.

   b. Shake until the KOH goes into solution.

   c. Dilute to volume with reagent water.

   d. Store at room temperature in a glass bottle. Reagent is stable 1 year.

   e. Equivalent weights and volumes are acceptable as long as the ratio remains constant.

10. Sodium Sulfate (Na_2SO_4) - Reagent grade or equivalent. Bake at 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

11. Acidified Sodium Sulfate

   a. Before use add 1.0 mL of concentrated sulfuric acid to 1 kg of baked sodium sulfate in a 2-L beaker and slurry with ethyl ether.

   b. Remove ethyl ether by placing the mixture on a steam bath.

c. Confirm the mixture is below pH of 4 by adding 1 g of the resulting solid to 5 mL of reagent water and checking the pH.

d. Store at approximately 130°C or in a desiccator.

e. Up to 100 g per sample is needed. (Equivalent weights and volumes are acceptable as long as the ratio remains constant).

12. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding Analysis #10401.

Preparation of Glassware

See 1-P-QM-PRO-9015475.

Calibration

Not applicable to this procedure.

Procedure

1. Use a wash bottles to rinse all glassware with 1:1 Acetone/Hexane.

a. This rinse reduces the amount of alkaline substances present preventing a reaction with the organic acids being extracted.

b. Be certain all solvent is completely evaporated before the glassware comes in contact with the samples or low recoveries of Dinoseb result.

2. If Sample Registration has pre-weighed the sample into a glass jar, add approximately 20 grams of acidified sodium sulfate, mix and proceed by transferring the sample from the jar into a stainless steel beaker. If the sample is not pre-weighed, weigh 30 to 30.5 grams of sample into a stainless steel beaker.

a. Record the initial weight and any comments about the sample in the extraction log.

b. Use of alternate weights is acceptable if necessary due to client specifications, amount of sample available, or sample matrix.

c. Process all tissues using Analysis #2487 prior to extraction.

d. The blank, Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD) (if applicable) are prepared by weighing 30 to 30.5 g of acidified sodium sulfate into a stainless steel beaker. Record the weight on the extraction log.

NOTE: The background, Matrix spike (MS), and Matrix Spike Duplicate (MSD) are performed on three separate aliquots of a field sample.

3. Add approximately 20 g of acidified sodium sulfate and mix thoroughly.

a. Use a disposable pipette to add a full pipette of concentrated hydrochloric acid to produce a slurry.

b. Allow the sample to sit for 15 minutes.

c. Check the pH using narrow range pH paper (1.0 to 2.5).

(1) If the pH is not below 2, add more acid until this pH is achieved.

(2) Alternatively, add 10 mL of phosphate buffer.

(3) This results in a wetter sample; additional acidified sodium sulfate is needed.

4. Use pipettes to add surrogate standards and matrix spiking solutions.

a. Surrogates: Add 1.0 mL Herb Surrogate to all samples, blanks, and spikes.

b. Spiking Solutions: Spiking solutions are added to the laboratory control sample (LCS), LCSD if applicable, MS and MSD samples. Spike 1.0 ml of Herb spike into LCS, LCSD (if applicable), MS and MSD. If directed by client, spike 1.0 ml of Hexachlorophene spike along with the Herb spike.

NOTE: This is changed to accommodate client-specific requirements as needed.

c. If a sample requires any special compounds in addition to the standard list, an appropriate spike containing those compounds is added at this time.

d. See Analysis #10401 for spike details.

e. See 1-P-QM-PRO-9015490 for storage and handling of spikes.

5. Use a solvent dispenser to add approximately 100 mL of 50% acetone in methylene chloride.

6. Set up the sonic probe as described in the manual. (See 1-P-QM-PRO-9015405.)

7. Immerse the tip of the sonic probe approximately 1 to 2 cm below the surface of the liquid in the beaker containing the sample and above the sediment layer.

8. Disrupt the sample using a medium tip at full output of 10 and a process time/timer of 1:30. (This is a total time of 1:30 pulse on and 1:30 pulse off.)

9. Remove the probe from the sample and decant the liquid through Fisher G6 filter paper into a vacuum flask.

10. Use a solvent dispenser to add 100 mL of fresh solvent to the sample and repeat Steps 7 through 9.

11. Use a solvent dispenser to add 100 mL of fresh solvent to the sample and repeat Steps 7 through 9 once more.

a. Pour the liquid and solids from the beaker onto the filter paper.

b. Rinse the beaker and filter paper with approximately 30 mL of 50% acetone in methylene chloride.

NOTE: Before placing the probe into another sample, wet a paper towel with reagent water and wipe the probe to remove any soil present from the previous sample. Use a wash bottle to rinse the probe with acetone to remove water.

12. Transfer the filtrate to a K-D apparatus and use a wash bottle to rinse the filter flask with approximately 30 mL of 50% methylene chloride and acetone to complete the transfer.

13. Add a boiling chip to the K-D apparatus and attach a Snyder column, wet the column with methylene chloride and concentrate to approximately 3 to 5 mL over a steam bath that is at 85° to 99°C. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. This steam bath temperature ensures concentration in a reasonable length of time.

14. Add 5 mL of 37% KOH solution. and 30 mL of reagent water to the KD apparatus.

15. Reflux on a steam bath at 80° to 85°C (60° to 65°C for all samples from North Carolina) for approximately 30 minutes (1 to 2 hours for samples from North Carolina). This steam bath temperature ensures concentration in a reasonable length of time.

a. All methylene chloride must be evaporated.

b. Allow to cool approximately 10 minutes before transfer to 125-mL separatory funnel to avoid bubbling in separatory funnel when methylene chloride is added.

16. Transfer the aqueous solution to a 125-mL separatory funnel. Use a wash bottle to rinse the K-D with reagent water to complete the transfer.

17. Shake the sample for 1 minute and 30 seconds with 50 mL of methylene chloride. Discard the methylene chloride (lower) layer.

18. Use a pipette to acidify the solution to pH <2 with 25% sulfuric acid. This requires approximately 5 mL of the acid solution.

19. Perform a 1-minute ethyl ether extraction by shaking the sample with 40 mL of ethyl ether.

a. Drain the aqueous layer into a clean, acid rinsed flask.

b. Place the ethyl ether (top) layer into an acid rinsed flask containing at least 30 grams of acidified sodium sulfate. (Sodium sulfate must be in a quantity so that the sample is completely dried). More sodium sulfate is added if necessary.

c. Return the aqueous layer to the separatory funnel for the next extraction.

20. Perform two additional 1 minute ethyl ether extractions by shaking the sample with 20 mL of ethyl ether.

a. Drain the aqueous layer into an acid rinsed flask.

b. Place the ethyl ether layer into the acid rinsed flask that contains 30 grams of acidified sodium sulfate.

c. Return the aqueous layer to the separatory funnel for the next extraction.

21. Allow the extract to remain in contact with the acidified sodium sulfate for a minimum of 2 hours while covered.

a. If the sodium sulfate is not free-flowing, add additional acidified sodium sulfate until all the water is removed.

b. Residual water hinders the methylation step and therefore must be removed with sodium sulfate before proceeding.

22. Transfer the extract into a K-D apparatus. Use a wash bottle to rinse the flask and funnel with two approximately 30-mL aliquots of ethyl ether to complete the transfer.

23. Add a glass bead to the K-D apparatus and attach a Snyder column, wet the column with ethyl ether and concentrate over a steam bath at 85° to 99°C until the apparent volume in the ampule reaches 1 mL.

This steam bath temperature ensures concentration in a reasonable length of time.

24. After the sample has cooled, use a squirt bottle to add approximately 0.5 mL of methanol.

25. Set up diazomethane generator and esterify the extract as described below:

a. Safety precautions

(1) Diazald is a carcinogen. Wear gloves at all times during this procedure. Perform esterification in a hood. Avoid inhalation of diazomethane.

(2) Avoid using etched or scratched glassware and ground glass joints. **Do not heat over 90°C (explosion may result).** The generator must be set up in a hood containing no electrical appliances or steam baths. The additional heat and electrical hazard must be avoided.

b. Diazomethane Generator Procedure

(1) Prepare the diazomethane solution. Reagents must be mixed in the following order.

(a) Place 5 grams of KOH in a 125-mL Erlenmeyer flask.

(b) Use a pipette to add 8 mL of reagent water.

(c) Allow the solution to cool, and then use a graduated cylinder to add 25 mL of reagent alcohol and a reagent pump to add 25 mL of ethyl ether.

(d) Fill at least two 40-mL vials with ethyl ether for rinsing the generator between samples.

(2) Just prior to starting the procedure (must be performed under hood) add approximately 3 grams of diazald to the KOH solution.

(a) Clamp the Erlenmeyer in place.

(b) The amount of diazald needed depends on the number of samples extracted. Use 3 g for eight to ten samples. If more than ten samples are extracted, prepare a second diazomethane solution for use during esterification.

(3) Fill a beaker with hot water (80° to 85°C) from the steam bath and hold under the Erlenmeyer containing the diazomethane solution. At the same time, hold a rinse vial of ethyl ether at the end of the generator.

(4) When the rinse vial begins to turn yellow, remove the vial and begin placing samples at the end of the generator.

(a) Using a rinse vial, rinse the generator between samples.

(b) Be sure each sample turns bright yellow before going on to the next sample. This is to ensure that esterification is complete.

(c) If the yellow color does not persist in all of the samples after methylation for the group is complete, remethylate the samples that are no longer yellow.

(5) After esterification, N-Evap the samples to 2 mL. **Do not use heat above 40°C.**

(6) Use a wash bottle to adjust the final volume to 10.0 mL with hexane.

Pour the extract into a clear 12-mL labeled vial.

(7) Florisil the sample as follows:

(a) Prepare a 2-gram florisil cartridge by rinsing two times with 3 to 5 mL of hexane. Discard the rinseate.

(b) Use a pipette to add 2 mL of extract to the cartridge. Elute to just above the meniscus using gravity. Collect eluent in a test tube.

(c) Pour 5 to 6 mL of 50% ethyl ether in hexane into the cartridge. Collect the rinse in the test tube.

(8) N-Evap the extract to just below 2 mL.

(9) Add 100 µL of Herb Internal Standard to a 2-mL volumetric flask.

Bring the extract to final volume of 2.0 mL in a 2-mL volumetric flask containing 100 µL of Herb Internal Standard with hexane.

(10) Use a disposable pipette to bottle the extract in a clear autosampler vial labeled with the sample number and an "F." The remainder of the unflorisiled extract is in the labeled 12-mL vial.

(11) The extracts are stored in a freezer at ≤-10° C and must be analyzed within 40 days of extraction.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

For each batch of samples extracted, a blank, LCS, MS and MSD must be extracted. If there is limited sample that prevents the preparation of the MS/MSD then an LCSD must be prepared instead.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed instead.

End of document

Version history

Version	Approval	Revision information
11	11.AUG.2016	

An error has happened on file: /D4Doc/formularer/2016/09/Quality Document # 1-P-QM-WI -9013472_11_4181.doc
Errortext: The remote server returned an error: (404) Not Found.

eurofins	Always check on-line for validity **Chlorinated Herbicides by 8151A in Solids by GC-ECD**	Level: **Work Instruction**
Document number: **T-PEST-WI9845**		
Old Reference: **1-P-QM-WI-9015158**		
Version: **13**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **29-AUG-2016**	Document users: **6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_Herbicide Chem**	Responsible: **5_EUUSLA_Pesticide Residue Analysis_Manager**

LIMS ID
Analysis DOD - 10401, 13434

Revision Log

Revision: 13		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Historical/Local Document Number	Reflects current analysis scans	Added 13434
Cross Reference	References not needed	Removed 1-P-QM-QMA-9015390 and 1-P-QM-QMA-9017309
Definitions	Common laboratory terms defined in higher level documents	Removed definitions

Revision: 13		**Effective Date:** This version
Personnel Training and Qualifications	Reference not needed	Removed reference 1-P-QM-QMA-9015390
Sample Collection, Preservation and Handling	Reflects current extract storage condition	Changed temperature of extract storage from (-10° to -15°C) to ≤-10°C
Reagents and Standards	Reflects current spiking standard solutions used by extraction department Reflects current standard storage conditions	Updated table. Revised temperature of storage of standards
Calibration	Reflects current process Old reference not needed	Removed changing septa prior to calibration. Removed old reference SOP-PP-031
Procedure 1.b	Old reference not needed	Removed old reference SOP-PP-011
Procedure 2	Reflects current practice	Added solution used for preparing dilutions.
Calculation	Old reference not needed	Removed old reference SOP-PP-040
Statistical Information/Method Performance	Reference not needed Reflects similar verbiage as other department SOPs	Removed old reference number SOP-PP-025 and 1-P-QM-QMA-9017309 Added similar verbiage for MDL study as other department SOPS
Quality Assurance/Quality Control	Old reference not needed	Removed old reference SOP-PP-002

Revision: 12		**Effective Date:** Aug 20, 2015
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Scope	Reflects current LOQs and target compounds.	Updated to current LOQs. Removed compounds that are not qualified (3,5-dichlorobenzoic acid, Acifluorfen, and Bentazon
Reagents and Standards	Reflects current standards	Updated DCAA SS stock, Herbicide intermediate, ICV intermediate, surrogate stock. Removed Hexachlorphene/Picloram intermediate. Added MS Hexachlorophene to be a separate standard.
Statistical Information/Method Performance	Clarification	Added information about the full MDL study
Quality Assurance/Quality Control	Enhancement	Added information on evaluating the internal standard.

Reference

1. *Test Methods for Evaluating Solid Waste*, SW-846 Method 8151A, December 1996.

2. State of Connecticut Department of Environmental Protection, Recommended Reasonable Confidence Protocols for Chlorinated Herbicides by SW-846 8151, version 2.0 July 2006.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #4181	Ultrasonic Extraction of Chlorinated Herbicides by Method 3550B/C in a Solid Matrix
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015493	QC Data Acceptability and Corrective Action
1-P-QM-PRO-9015494	Interpretation of Chromatographic Data
1-P-QM-PRO-9015496	Monitoring QC Data Acceptance Limits
1-P-QM-PRO-9015498	Setting Up Single Component Initial Calibrations
1-P-QM-PRO-9015501	Common Equations Used During Chromatographic Analyses

Scope

This method is used for identifying and quantitating the following chlorinated herbicides in soils, sediments, and solids.

Compound	Limit of Quantitation (µg/kg)	
2,4 – D	36	
2,4 – DB	17	
2,4 - DP (Dichloroprop)	18	
2,4,5 – T	1.7	
2,4,5 - TP (Silvex)	1.7	
Dalapon	90	
Dicamba	12	
Dinoseb	24	
Hexachlorophene	24	Special request required
MCPA	2500	
MCPP (Mecoprop)	2500	
Pentachlorophenol	1.7	
Picloram	40	Special request required

Limits of Quantitation (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

See Analysis #4181 for the extraction preparation.

Basic Principles

The compounds of interest are extracted with 1:1 methylene chloride:acetone from an acidified portion of soil (pH less than 2) using sonication. The extract is hydrolyzed and interfering compounds like chlorinated hydrocarbons and phthalates are removed by a solvent wash. After acidifying the extract once more, the compounds are extracted with ethyl ether and are converted to their methyl esters using diazomethane as the derivatizing agent. The methyl esters are determined using gas chromatography with an electron capture detector. A florisil cleanup is performed to eliminate matrix interferences that introduce large, unresolvable peaks in the chromatogram. A dilution is required if interferences such as chlorinated acids and phenols are present. Refer to 1-P-QM-PRO-9015477 for more details on this cleanup procedure.

Interferences

An electron capture detector is very sensitive to compounds that contain halogens and responds to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur. Plastic must not be used during the extraction or analysis to prevent phthalate contamination. Glassware must be scrupulously cleaned. All of these interferents can introduce large, unresolvable peaks into the chromatogram. Florisil cleanup is used to reduce other organics which can interfere (polar compounds). Additionally, the extraction incorporates a solvent wash step to remove potential interfering organic compounds.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Safety glasses must be worn around the GC where solvents and extracts are handled.

All GC vials are disposed of in the designated waste container in the lab, then subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the lab, then emptied into the lab-wide waste facility.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs.

Sample Collection, Preservation, and Handling

Unpreserved samples must be collected in wide-mouth glass jars with PTFE-lined lids and stored at 0° to 6°C, not frozen. The holding time is 14 days from collection to extraction. The extracts must be stored at $\leq$-10° C and analyzed within 40 days of extraction.

Apparatus and Equipment

1. HP7890 gas chromatograph fitted with electron capture detector, or equivalent
2. Columns:
   a. Phenomenex ZB-XLB 30 m × 0.32 mm × 0.25 µm
   b. Phenomenex ZB-35 30 m × 0.32 mm × 0.25 µm
3. Integrating system such as ChromPerfect from Justice Innovations or equivalent
4. Various sizes of Class A volumetric pipettes, flasks, and syringes.

Reagents and Standards

A. Reagents

1. Hexane, pesticide grade for autosampler vials. Stored at room temperature.

2. UPC (ultra pure carrier) nitrogen for detector make up

3. UPC helium for carrier gas

4. UPC hydrogen, bottled or from a generator

B. Standards

1. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

2. All standards are prepared using Class-A volumetric flasks, pipettes, and syringes.

3. All standards are stored in labeled vials or flasks in a freezer at ≤-10° C.

4. Herb stock – Acid herbicide stock for calibration standards: Accustandard catalog #S-12214-R5 in methanol (concentrations vary per compound).

5. DBOFB stock – 4,4'-dibromooctafluorobiphenyl Ultra PPS-170 at 1000 µg/mL used as internal standard.

6. DBOFB Intermediate – Dilute 1.0 mL of DBOFB stock into 50 mL of acetone. This solution is stable for 6 months.

7. Working DBOFB IS diluting solution – Dilute 5.0 mL of the DBOFB intermediate into 100 mL of hexane. This solution is used to make sample dilutions. This solution is stable for 6 months.

8. DCAA SS stock – 2,4 dichlorophenylacetic acid (DCAA) non–methylated stock. Ultra PPS–162 at 5000 µg/mL.

9. Herbicide intermediate – Dilute 1.0 mL of acid herbicide stock, 0.02 mL of hexachlorophene stock, 0.04 mL of picloram stock, and 0.02 mL of DCAA SS stock into 10 mL of ethyl ether. This solution is methylated following procedure outlined in Analysis #4181 prior to making the calibration standards. This solution is stable for 6 months.

10. MS stocks:

a. Herbicide mix in the acid form for the matrix spiking solution: Ultra cat #HBM-8150A in methanol (concentrations vary per compound).

b. PCP stock – Accustandard cat #APP-9-176-D-20X at 2000 µg/mL in methylene chloride.

c. Dinoseb fortification stock – Restek cat #32251 at 1,000 µg/ml in methanol.

d. Picloram stock – Restek cat #32265 at 1,000 µg/ml in methanol.

e. Hexachlorophene stock – Restek cat #EPA-1125 at 1,000 µg/ml in acetone.

11. Hexachlorophene stock – Restek cat #31811 at 2,000 µg/ml in methylene chloride.

12. Picloram stock – Restek cat #32265 at 1,000 µg/ml in methanol

13. ICV Stocks:

a. Acid herbicide mix - Supelco cat # 46861-U in methanol (concentrations vary per compound)

b. Hexachlorophene – Accustandard cat #APP-9-116 at 100 µg/l in methanol

14. ICV Intermediate – Dilute 1 mL of acid herbicide stock, 0.5 mL of hexachlorophene stock, and 0.02 mL of DCAA SS stock into 10 mL. This solution is methylated following procedure outlined in Analysis #4181 prior to making the working standard.

15. Surrogate Stock (SS) – Chem Service, cat# S-10536B5-5m containing 2,4-Dichlorophenylacetic acid (DCAA) in acetone.

16. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. HERB 1), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 40 fold of the initial calibration level.

17. Prepare the spiking solutions using the prep scheme in the table below:

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description
MS	Herb mix Stock	2.5	100	Methanol	Herb Spike
	PCP Stock	0.05			

	Dinoseb Fortification Stock	0.3			
	Picloram Stock	0.25			
	Hexachlorophene	0.12			
IS	4,4 DBOFB Solution	1	50	Hexane	Herb Internal Std
SS	SS DCAA Stock	1.0 ml	1000ML	Acetone	Herb Surrogate Standard
IBLK	SS Stock	0.88	200	Hexane	Instrumen Blank Std
	DBOFB stock	0.20			

GC instrument Conditions

Instrument setup (Primary and Confirmation):

Detector:	ECD
Detector Temperature:	300°C
Makeup Gas:	N2 at 30 mL/min for Varian ECDs, 55 mL/min for HP ECDs
Injection Size:	2 µL, direct injection
Injector Temperature:	250°C
Oven Temperature:	50°C, hold 0.5 min, 25°C/min to 100°C, 12°C/min to 310°C, hold 2 min
Carrier:	Hydrogen at 10 psi (Helium is a substitute.)

The conditions listed above are optimum but are changed to improve the linearity, sensitivity, and chromatography on each GC system. A Merlin microseal can be used in place of a traditional septum.

Calibration

1. Prior to starting a new calibration, fill the autosampler rinse vials with clean solvent or replace vials themselves if they appear to be dirty.

2. Prepare a sequence as follows:

    1. Conditioner
    2. IBLK
    3. Herb Level 1
    4. Herb Level 2
    5. Herb Level 3
    6. Herb Level 4
    7. Herb Level 5

8. Herb level 6 (optional)
9. MDHEX
10. ICHBX (ICV)
11. Blank
12. LCS
13. 1234567
14. 1234567ms
15. 1234567msd
16. – 20. Continue with samples
21. Herb Level 3
22. – 31. Ten samples
32. Herb Level 3

Continue running groups of 10 samples followed by an Herb level 3 standard between sample groups.

3. The conditioner injection is usually a standard or sample that has already been injected.

a. Any number of conditioners can be used prior to the start of the run – one is shown here as an example.

b. It is used to prime the system and is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

c. Hexane blanks are run to allow the GC to go through some temperature program runs and/or to check the cleanliness of the system when needed.

4. The instrument blank (IBLK) is injected after the conditioners but before the initial calibration.

a. It is used to determine that the instrument is free of background noise or contamination.

b. IBLK may also be run with the continuing calibration standards – this is optional, but is frequently requested for projects.

5. Initial Calibration (ICAL)

a. The system is calibrated using a minimum of five concentration levels.

b. An internal standard calibration is used with average response factor (AVGRF) for all analytes where the %RSD is ≤20%.

(1) If the average of the %RSDs of all compounds in the initial calibration standard is ≤20%, the AVGRF is used for all compounds in the ICAL when needed.

(2) Alternatively, when these criteria are not met, a calibration curve must be used.

c. When using a calibration curve, a linear fit must be tried first.

(1) Use the linear fit if the correlation coefficient is >0.99.

(2) However, if the correlation coefficient is <0.99, a quadratic fit must be tried. A 6−point calibration must be run to use quadratic.

d. For either curve type, extrapolate or force zero is not allowed.

e. See 1−P−QM−PRO−9015498 for details on using Chrom Perfect for setting up single component calibration files.

f. Ensure all peaks in the standard are labeled properly and the scaling of the plot is such that concentrations at the MDL exhibit a peak about 2 to 3 mm in height.

g. Be sure all peaks on the MDHEX standard are integrated by the data system.

6. Initial Calibration Verification (ICV)

a. Verification of the calibration curve is performed using the ICV mixtures injected directly after the full ICAL.

b. The % difference (%D) of the concentrations for each analyte must be within ±15%D of the nominal concentration for the curve to be used for sample analysis.

7. Continuing Calibration Verification (CCV)

a. A CCV standard is analyzed after every ten injections using the level 3 calibration standard.

b. The CCV between samples must exhibit a response at ±15%D for each compound, or the average of the %Ds must be within ±15% for the standard to be compliant on at least one of the two columns used for analysis.

c. The concentration calculated for the CCV injection is compared to the nominal concentration.

d. Samples must be bracketed with compliant standards.

(1) Exception: If, however, the standard following a sample is outside the ±15% but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected.

(2) If confirmation of target analytes is needed, then the second column should meet the 15% CCV criteria, as well as all ICAL criteria.

8. If an instrument blank (IBLK) is injected after the CCV, it must be evaluated as a water matrix against the water MDL/LOQs.

a. The IBLK must not have any target compounds above the reporting limits.

b. If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(1) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be reinjected after another compliant IBLK.

(2) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up the instrument.

9. Retention time (RT)

a. RT windows are established as 3× the standard deviation determined over a 72−hour period, or at no less than ±0.03 min, applied to the mid-point initial calibration standard.

b. If the RTs for a CCV fall outside the windows, update the midpoint RT using that standard.

(1) Save this under an appropriate name to indicate an update has occurred.

(2) All subsequent continuing standards run within a 24−hour period must fall within this window.

(3) RTs cannot be updated more than once per day.

(4) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Retention times of peaks in the samples are compared to the standard RT windows.

a. Peaks that are present on both columns are quantitated and the high value is reported unless there are chromatograph anomalies.

b. See 1−P−QM−PRO−9015494

2. Samples that contain levels of analytes above the highest level calibration standard must be diluted and reanalyzed.

When preparing dilutions, add sufficient internal standard to maintain the same 100−µg/L final concentration. The Working DBOFB IS diluting solution is used when preparing dilutions. See standard section.

3. If data is reported using the grand mean for the ICAL or CCV, the following comment(s) will be added to the report:

a. 1571 – The % difference for the calibration verification standard is outside the ± criteria for the analyte(s) listed below. Since the average of the % difference values meets the criteria, the results are reported.

b. 1572 - The % relative standard Deviation for the initial calibration or the analyte(s) listed below is above the 20% criteria. Since the average of the RSD values for all calibrated compounds meets the criteria, the average response factor calculation was used.

c. **NOTE:** Use of the average of the %Ds (grand mean) is not permitted for samples from South Carolina and may not be approved for specific client projects.

Calculations

$$SampleConc.(\mu g/kg) = ExtractConc. \times \frac{DF \times FV}{IW} \times Conc.Internal\,Standard$$

Where:

FV = Final volume = 100 mL

IW = Initial weight = 10 g

DF = Dilution factor

1. Linear curve

$$ExtractConc.(\mu g/L) = \frac{\frac{Peak\,Height}{Internal\,Standard\,Height} - Y - intercept}{slope}$$

2. Average response factor (AVG RF)

The calculation performed by AVG RF is the same as above except the extract concentration is calculated as follows:

Where:

$$ExtractConc.(\mu g/L) = \frac{\left(\frac{Peak\ height}{internal\ standard\ height}\right)}{AVG\ RF}$$

Where:

$$AVG\ RF = (RF\ Calib\ 1 + RF\ Calib\ 2 + \ldots + RF\ Calib\ 5) / 5$$

$$RF = \frac{Standard\ Peak\ Height}{Internal\ Peak\ Height} / \frac{Standard\ Concentration(mg/L)}{Conc.\ Internal\ Standard}$$

Also see 1-P-QM-PRO-9015501 for more details on calculations regarding the calibration and QC samples.

Statistical Information/Method Performance

The QC acceptance limits for LCS, MS/MSD and surrogates are established according to 1-P-QM-PRO-9015496.

Initially, perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire extraction and analysis procedure. The results are tabulated using an Excel spreadsheet. Compare and pool results to determine the final reporting MDL. An MDL study or verification of the MDL is required each year. NELAC allows for an annual verification of the MDL in lieu of a full MDL study. The department supervisor maintains annual study data. Updates to the LIMS are made as need by the QA Department and only as directed by the manager. Update the department database via a download from the LIMS.

Quality Assurance/Quality Control

A sodium sulfate blank and LCS are analyzed with each group of samples. MS and MSD are analyzed with each batch of 20 samples as long as there is ample volume. An LCSD must be performed if an MS/MSD cannot be done.

The spiking solutions contain all analytes of interest. A surrogate standard of 2,4-dichlorophenyl acetic acid (DCAA) is added to each sample, blank, and spike to monitor the efficiency of the extraction and the

operation of the autoinjector. An internal standard is added to each sample, blank, and spike. The internal standard (DBOFB) response for each sample and QC is evaluated by comparing the response in the sample to the response of the nearest check standard. Since there is no criteria listed in the method, an advisory window of 50 – 150 is used.

If more than 20 samples are prepared in a day, then an additional batch with QC must be extracted. If any client, agency or state has more stringent QC or batch requirements, these must be followed instead.

1-P-QM-PRO-9015493 outlines the QC acceptability criteria and corrective action.

See Appendix I for the specific CT RCP criteria.

Appendix I

CT RCP Requirements

No use of grand mean for initial and continuing calibration evaluation.

If any sample needs a comment about out of spec data, comment 2510 must be put on the sample first, followed by our comment, so that the RCP form generates properly from the LIMS.

Comment about any raised limits (does not need precursor comment)

Need instrument blanks after every CCV set.

If there is >40% difference between column A and B, a comment must be placed on the analytical report. There are two comments for this: 1570 says the higher result was reported, 1569 says the lower result was reported. Use these on the analytical report as they apply.

The LCS recoveries must be between 40 -140%.

The MS/MSD recoveries must be between 40-140% with the RPD ≤ 30%.

The surrogate recovery must fall between 30 – 150%.

End of document

Version history

Version	Approval	Revision information
13	29.AUG.2016	

eurofins	Always check on-line for validity **Extraction of Chlorinated Herbicides in a Water Matrix by SW-846 8151A**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10919**		
Old Reference: **1-P-QM-WI -9015078**		
Version: **15.1**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **28-DEC-2016**	Document users: **5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Herbicide Waters and , 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_Herbicide Chem**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID
Analysis DOD - 0816, 11110, 11111

Revision Log

Revision: 15		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1 -P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Analyses were deactivated	Removed analyses 5593 and 5300
Definitions	Common terms defined in higher level document	Removed section and defined first use of each acronym in document
Personnel Training and Qualification	Reflects current process	Added detail regarding the performance of IDOCs and DOCs

Revision: 15	**Effective Date:** This version	
Sample Collection, Preservation, and Handling	Reflects current process Reflects current process	Revised the temperature storage requirements for samples and extracts Added type of collection bottle
Reagents and Standards	Reflects current process	Added the word approximately to describe the temperature for baking sodium sulfate and sodium chloride
Procedure 9.a	Clarification	Described rinsing of sample bottle with methylene chloride
Procedure 13	Reflects current process	Added step for visual confirmation of adequate phase separation during shake
Procedure 18	Reflects current process	Added the word approximately to describe wait time
Procedure 23	Reflects current process	Added language regarding swirling of extract and sodium sulfate to ensure complete absorption of residual water
Procedure 25	Enhancement	Clarified how to achieve a reasonable length of time for concentration
Procedure 29	Reflects current process	Revised final volume accuracy to 10.0 mL
Procedure 30	Reflects current process	Revised final volume accuracy to 2.0 mL

Revision: 14	**Effective Date:**	Apr 12, 2013
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Basic Principles	Correction as per Method 8151A section 7.3.3.1 Correction as per Method 8151A section 7.3.4	Changed >12 to =12. Changed <2 to =2.
Reagents and Standards	Analysis was deactivated	Removed analysis 5593.
Procedure 8.	Correction as per Method 8151A section 7.3.3.1	Changed >12 to greater than or equal to 12 and changed =12 to less than 12.
Procedure 14.	Correction as per Method 8151A section 7.3.4	Changed <2 to less than or equal to 2 and changed =2 to greater than 2.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8151A, December 1996.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0952, 10407	Analysis of Chlorinated Herbicides by 8151A in Water

1-P-QM-PRO-9015475	Glassware Cleaning for Organic Extractions
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Scope
This method is for the extraction of chlorinated herbicides in water and wastewater using SW-846 8151A.

Basic Principles
An aliquot of the sample is placed into a separatory funnel with sodium chloride and a surrogate standard is added to monitor recovery. The pH is adjusted to =12 and the sample is allowed to sit for 1 hour with occasional shaking. The sample is then extracted with methylene chloride and the solvent discarded. The pH is adjusted to =2 and the sample is serially extracted with ethyl ether. The ether fractions are combined and placed in a flask containing acidified sodium sulfate. The mixture is allowed to sit for at least 2 hours. Then the solvent is transferred to a K-D apparatus and concentrated to 10 mL. Methanol is added and the sample is subjected to esterification. The extract is then concentrated to 2 mL using nitrogen blow down technique. Hexane is added to adjust the final volume and the extract is florisiled.

Procedural changes to use a reduced sample aliquot while maintaining the default reporting limits are permitted as long as the reagent aliquots are reduced proportionally and a quad study is performed and on file.

Reference Modifications
1. Surrogate and matrix spiking solutions are not added before the transfer to the separatory funnel for several reasons:

a. Samples must be poured from the amber bottles to determine the matrix and volume of sample to use for each extraction.

b. Many sample bottles have no headspace and there is no room to add surrogate to the sample in the bottle.

c. Due to the volume of samples extracted, a separate graduated cylinder for each sample is unrealistic.

d. To maintain consistency with all extractions, no samples are spiked in the bottle or graduated cylinders.

2. The extraction as explained in the Procedure through Step 13 is sufficient to force the herbicide salts into the aqueous solution. Additional extraction with methylene chloride has been found to be unnecessary.

3. The sulfuric acid is not chilled prior to addition in the Procedure Step 14. Since the acid is added using a reagent pump, there is minimal chance of contact.

4. 30 g of sodium sulfate is used to ensure **all** H_2O is absorbed in the Procedure Step 19.

5. Nitrogen gas is not used to bubble the diazomethane through the sample in Procedure Step 27.b.(4). Instead, the diazold solution is gently heated using a beaker of hot water.

6. Florisil cleanup is performed in the Procedure Step 30 to remove interferences that inhibit or negatively impact the determination of herbicide compounds.

Interferences

Impurities in solvents, reagents, glassware, or other hardware used in sample processing lead to interferences with the method. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Always add acid to water to reduce fuming. The sulfuric acid and sodium hydroxide solutions get hot when prepared. Always wear gloves when handling these solutions.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Always wear gloves when handling the diazald and avoid inhaling diazomethane gas. Both are extremely toxic, severely irritating, and have been cited as carcinogens. See specific safety instructions for this procedure listed in the esterification section.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus due to the high temperatures.
All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers. All waste generated from esterification must be placed in a beaker in the hood and must be added to the solvent waste stream in the lab-wide disposal facility.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the technicians training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the extraction and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, or one blind sample.

Sample Collection, Preservation, and Handling

Samples must be collected in 1 liter amber glass bottles and stored at 0° to 6°C, not frozen, prior to extraction. Extraction must be started within 7 days of collection. The extract is stored in the freezer at =-10 °C.

Apparatus and Equipment

1. Separatory funnels, 2-L

2. Kuderna-Danish (K-D) assembly with appropriate ampule for concentrating the solvent used during concentration

3. Beakers – Assorted

4. Water bath

5. Filter paper – Whatman #3 or equivalent

6. N-Evap with nitrogen supply

7. pH meter or paper – Assorted ranges

8. Diazomethane generator

9. Balance – Capable of weighing to 0.01 g

10. Wash bottles

11. Scriber

12. Graduated cylinders – Assorted sizes – Class A

13. Automatic shaker – Capable of holding 2000 mL separatory funnels

14. Pipettes – Class A, assorted sizes

15. Syringes – Assorted sizes

16. Micropipetter

17. Reagent pump

18. Erlenmeyer flasks – 500 mL

19. Boiling beads – glass

20. Pipettes – Disposable

21. Test tubes

22. TCLP extraction fluid – delivered weekly by the leachate department

Reagents and Standards

1. Methylene chloride (CH_2Cl_2) – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

2. Acetone – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

3. Hexane – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

4. Ethyl ether, high purity, nonpreserved – Use peroxide test strips to ensure it is free of peroxides. Store at room temperature. Follow manufacturer's expiration information.

5. Sulfuric acid (H_2SO_4) – Baker Instra-analyzed or equivalent. Store at room temperature for up to 1 year.

To prepare 12N solution: Add 588.1 ± 1.0 g H_2SO_4 to a 1000 mL volumetric flask containing approximately 400 mL of reagent water. Dilute to 1000 mL with reagent water.

6. 6N sodium hydroxide (NaOH) – Lab Chem or equivalent. Store at room temperature for up to 1 year.

7. Diazald (N-methyl-N-nitroso-*p*-toluenesulfonamide) - Follow manufacturer's storage and expiration information.

8. Reagent water – water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets this criterion. If the reagent water does not meet the requirements, see your supervisor for further instructions.

9. Alcohol GR. Store at room temperature for up to 1 year.

10. Sodium chloride (NaCl) – Bake at approximately 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

11. Sodium Sulfate (Na_2SO_4) – Sodium sulfate (Na2SO4) – Granular anhydrous reagent grade or equivalent. Bake at approximately 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

12. Sodium sulfate, acidified – Up to 300 g per sample is needed.

a. Use a pipette to add 1 mL of concentrated sulfuric acid to a beaker containing 1000 g of baked sodium sulfate and slurry with ethyl ether. Remove ethyl ether by placing the mixture on a steam bath.

b. Confirm the mixture is below pH of 4 by adding 1 g of the resulting solid to a vial containing 5 mL of reagent water and check pH.

c. Store at 130°C or in a dessicator for up to one year.

13. Potassium Hydroxide (KOH) – ACS Certified, Fisher Chemical or equivalent. Store at room temperature for up to 5 years.

14. 50% Ethyl Ether in Hexane – Make desired amount by mixing equal parts of Hexane and Ethyl Ether. Store solution at room temperature for up to one year.

15. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical Analysis #0952, 10407

Preparation of Glassware

See 1-P-QM-PRO-9015475 (SOP-OE-001).

Calibration

Not applicable to this procedure.

Procedure

1. Use a wash bottle to rinse all glassware with acetone.

a. This rinse reduces the amount of alkaline substances present that react with the organic acids being extracted.

b. Be certain all acetone is completely evaporated before the glassware comes in contact with the samples to avoid low recoveries of Dinoseb.

2. Determine the volume of sample to be used for each extraction.

a. Analysis 10407

(1) Use one bottle (1 liter) unless the matrix is poor (thick, lots of sediment, extremely foul odor).

(2) If using reduced volume of sample due to matrix problems, reduced volume aliquots are 500, 200, or 100 mL.

b. Analysis 0952

(1) If at least 200 mL of sample is available, use 50 mL of sample.

(2) If the sample also requires a matrix spike, use 50 mL for the spike sample.

(3) If <200 mL is available, use ½ of the volume in the bottle.

(4) If the sample is not water soluble, use 1 mL.

c. If uncertain of the volume to extract for any sample, ask your supervisor.

3. Weigh approximately 250 g of NaCl and add to each separatory funnel.

4. Once the volume of sample is determined, measure each sample.

a. For samples that use one full bottle.

(1) Etch the outside of the bottle with a scriber at the meniscus of the sample volume.

(2) Shake the bottle vigorously and then pour the contents into a 2-L separatory funnel.

(3) Record any comments about the samples in the extraction log.

b. For all samples that use a specified volume.

(1) Shake each bottle vigorously, and then use a clean graduated cylinder to measure the volume.

(2) Transfer the sample to a graduated cylinder.

(3) Record the sample volume and any comments about the samples in the extraction sheet and transfer to the 2-L separatory funnel.

(4) Use a wash bottle to rinse the graduated cylinder with methylene chloride. After the 1 hour in Procedure 8 Step b., add the rinseate to the separatory funnel. If <1000 mL of sample is used, add enough reagent water to bring the volume in the separatory funnel to 1 L.

c. The blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) (if applicable)

(1) For analysis 0952: Prepared by measuring 200 mL of extraction fluid into the separatory funnel.

(2) For Analysis 10407: Prepared by using 1 L of reagent water measured into the separatory funnel.

d. The background, matrix spike (MS), and matrix spike duplicate (MSD) are prepared using three separate aliquots of a field sample.

5. Use a reagent pump to add 17 to 25 mL of 6N NaOH to each separatory funnel.

6. Use pipettes to add surrogate standards and spiking solutions to the aqueous sample in the separatory funnel.

a. The standard must drip directly into the aqueous sample without touching the glass side of the separatory funnel to avoid poor recoveries.

b. Surrogates –

For 0952 &10407 - Add 1.0 mL herb surrogate to all samples, blanks, and spikes.

c. Spiking solutions

(1) For analysis 0952 – Add 1.0 mL TCLP herb spike (this must be measured using a syringe or micropipette) to the LCS, LCSD (if applicable), MS and MSD.

(2) Analysis 10407– 1.0 mL herb spike and 1.0mL MCPA fortification mix.

(3) If the client requires an additional LCS/LCSD for Hexachlorophene,- spike using 1.0 mL hexachlorophene spike

(4) Spikes are changed to accommodate client-specific requirements as appropriate.

d. If a sample requires any special compounds in addition to the standard list, an appropriate spike containing the compounds is added at this time.

e. See 1-P-QM-PRO-9015490 (SOP-OE-017) for storage and handling of spikes.

7. Shake the sample on an automatic shaker for approximately one minute to dissolve the NaCl.

8. Check the pH using pH paper to ensure it is greater than or equal to 12.

a. If the pH is less than 12, add additional 6N NaOH until the pH is =12.

b. Allow sample to sit for 1 hour.

9. If the original sample bottle is empty.

a. Use a reagent pump to measure 60 mL of methylene chloride and rinse the sample bottle by capping and inverting bottle several times.

b. Add the solvent to the separatory funnel.

c. After the bottle is rinsed with methylene chloride, fill the bottle to the marked level with water and transfer the water to a graduated cylinder to determine the initial volume. Alternatively, weigh the empty bottle and tare the balance.

d. Fill the bottle to the marked level with water and place the bottle onto the tared balance. This weight rounded to a whole number is the initial sample volume.

e. Record the initial volume on the extraction sheet.

10. If the sample container is not empty, use a reagent pump to measure 60 mL of methylene chloride and add the solvent directly to the separatory funnel.

11. Cap the funnel, invert it, and vent immediately.

12. Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

NOTE: Shaker speeds vary greatly between instruments; the proper setting is marked on each.

13. Allow the phases to separate for approximately 10 minutes. The time required for extracts to set undisturbed is based upon visual confirmation that the layers are adequately separated. Additional time may be necessary for samples with unusual high density (i.e. high salt content). Open the stopcock and drain the methylene chloride (bottom layer) into a waste beaker and discard.

14. Use a reagent pump to carefully add 15 to 20 mL of 12N H2SO4 to each funnel.

    a. Shake to mix.

    b. Check the pH using pH paper to ensure it is less than or equal to 2.

    c. If the pH is greater than 2, add additional 12N H2SO4 until the pH is =2.

15. Use a reagent pump to add 120 mL ethyl ether to the separatory funnel.

16. Cap the funnel, invert it, and vent immediately.

17. Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

18. Allow the phases to separate for approximately 10 minutes.

19. Open the stopcock and drain the aqueous layer (bottom layer) into an acid rinsed 1-L beaker.

Drain the ethyl ether layer into a 500-mL Erlenmeyer flask that contains approximately 30 g of acidified sodium sulfate.

20. Pour the aqueous layer back into the separatory funnel.

21. Use a reagent pump to add 70 mL of ethyl ether into the separatory funnel. Extract the sample as listed in Procedure Steps 17 through 20 venting only if necessary.

22. Perform a third extraction using 70 mL of ethyl ether and follow Procedure Steps 17 through 19.

23. Allow the extract to remain in contact with the acidified sodium sulfate for a minimum of 2 hours while covered. Residual water hinders the methylation and results in low recoveries. Periodically swirl the Erlenmeyer flask containing the extract and acidified sodium sulfate to ensure the sodium sulfate is free flowing and that residual water is being absorbed.

NOTE: If the sodium sulfate is not free-flowing, add additional acidified sodium sulfate until all the water is removed.

24. Pour the extract into a K-D apparatus.

a. Break up the sodium sulfate with a glass rod.

b. Use a wash bottle to rinse the flask and funnel with two approximately 30-mL aliquots of ethyl ether to complete the transfer.

25. Add a glass-boiling bead to the K-D apparatus and attach a Snyder column. Wet the column with ethyl ether and concentrate over a steam bath at 80° to 85°C until the apparent volume in the ampule reaches 1 mL. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 5-10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

NOTE: For samples from **North Carolina** use a temperature of 60° to 65°C.

26. After the sample has cooled, use a squirt bottle to add approximately 0.5 mL of methanol.

27. Set up diazomethane generator and esterify the extract as described below:

a. Safety precautions

(1) Diazald is a carcinogen. Wear gloves at all times during this procedure.

(2) Perform esterification in a hood with the sash down. Additional protection, such as a safety shield, is recommended. Avoid inhalation of diazomethane.

(3) Avoid using etched or scratched glassware and ground glass joints**.**

(4) **To avoid explosion do not heat over 90°C.**

(5) All glassware used in this procedure must be taken directly to wash room personnel, not placed on cart of dirty glassware.

(6) The generator must be set up in a hood containing no electrical appliances or steam baths. Additional heat and electrical hazard must be avoided.

b. Diazomethane Generator Procedure

(1) Prepare the KOH solution.

Reagents must be mixed in the following order:

(a) Place 5 g of KOH in a 125-mL Erlenmeyer flask.

(b) Use a pipette to add 8 mL of reagent water.

(c) Allow the solution to cool.

(d) Use a graduated cylinder to add 25 mL of reagent alcohol.

(e) Use a reagent pump to add 25 mL of ethyl ether.

(2) Prepare the diazomethane solution under a hood and just prior to starting the procedure:

(a) Add approximately 3 g of diazald (5 or 6 pipette fulls) to the KOH solution.

(b) The amount of diazald needed depends on the number of samples extracted. Use 3 g for eight to ten samples. If more than ten samples are to be extracted, prepare a second diazomethane solution for use during esterification.

(b) Clamp the Erlenmeyer in place.

(3) Fill two 40-mL vials with ethyl ether for rinsing the generator between samples.

(4) Fill a plastic beaker with hot water from the steam bath and hold under the Erlenmeyer containing the diazomethane solution. At the same time, hold a rinse vial of ethyl ether at the end of the generator.

(5) When the rinse vial begins to turn yellow, remove the vial and begin placing samples at the end of the generator.

(a) Use a rinse vial to rinse the generator between samples.

(b) Be sure each sample turns bright yellow before going on to the next sample. This ensures that esterification is complete.

(c) If the yellow color does not persist in all of the samples after methylation is complete, remethylate the samples that are no longer yellow.

28. After esterification, N-Evap the samples to 2 mL.

29. Use a wash bottle to bring the extract back up to 10.0 mL with hexane.

30. Florisil the sample as follows:

a. Prepare a 1-g florisil cartridge by rinsing twice with approximately 3 mL of hexane for each rinse. Discard the rinse.

b. Use a pipette to add 2 mL of extract to the cartridge.

c. Elute to just above the meniscus at a flow rate of 5 mL per minute. Collect eluate in a test tube.

d. Pour approximately 4 to 6 mL of 50% ethyl ether in hexane into the florisil cartridge. Elute the florisil cartridge by slowly adding the solvent mixture to the cartridge. Collect the rinses in the test tube.

e. Add 100 µL of herb internal standard to each sample, blank, and spike sample. Use a dedicated syringe or micropipettes for this purpose.

f. N-Evap the extract to just below 2 mL. Use a disposable pipette and a 2-mL volumetric flask to bring to exactly 2.0 mL with hexane. Mix thoroughly.

g. Use a disposable pipette to bottle the florisiled extract into two clear autosampler vials labeled with the sample number.

31. Put the remainder of the extract in a 12-mL vial.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
A batch is defined as the samples to be extracted on a given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

For each batch of samples extracted, a blank, LCS (extraction fluid blank spiked with compounds to be determined carried through the entire procedure), MS, and MSD must be extracted. If there is limited sample preventing the preparation of the MS/MSD, an LCSD must be prepared instead. Also, if the batch contains only field or equipment blank samples, the LCS/LCSD QC pairing must be used.

See the GC analysis methods for specifics on compounds in the matrix spikes and surrogates.

End of document

Version history

Version	Approval	Revision information
15	28.MAR.2016	
15.1	28.DEC.2016	

An error has happened on file: /D4Doc/formularer/2016/09/Quality Document # 1-P-QM-WI -9015078_15_0816.doc
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eurofins	Always check on-line for validity **Analysis of Chlorinated Herbicides by 8151A in Water**	Level: **Work Instruction**
Document number: **T-PEST-WI9202**		
Old Reference: **1-P-QM-WI-9015088**		
Version: **14**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **26-AUG-2016**	Document users: **6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_Herbicide Chem**	Responsible: **5_EUUSLA_Pesticide Residue Analysis_Manager**

LIMS ID

Analysis DOD - 0952, 10407

Revision Log

Revision: 14		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Reference	Reflects current reference to Connecticut work	Added Reference for State of Connecticut Department of Environmental Protection RCPP
Cross Reference	References not needed	Removed 1-P-QM-QMA-9015390 and 1-P-QM-QMA-9017309
Definitions	Common laboratory terms defined in higher level documents	Removed definitions

Revision: 14	**Effective Date:** This version	
Basic Principles	Old reference not needed	Removed old reference SOP-OE-004
Personnel Training and Qualifications	Reference not needed	Removed reference 1-P-QM-QMA-9015390
Reagents and Standards	Reflects current extract storage condition	Changed temperature of extract storage from (-10° to -15°C) to <-10°C
Reagents and Standards 18.	Reflect current naming and spiking solutions used by extraction department.	Updated table
Calibration	Old reference not needed. Reflects current process	Removed old reference SOP-PP-031 Removed changing septa prior to calibration.
Procedure	Old reference not needed Reflects current practice	Removed old reference SOP-PP-011 Added using Working DBOFB IS diluting solution to perform dilutions.
Calculations	Old reference not needed	Removed old reference SOP-PP-040
Statistical Information/Method Performance	References not needed	Removed reference 1-P-QM-QMA-9017309 and old reference SOP-PP-025
Quality Assurance/Quality Control	Old reference not needed	Removed old reference SOP-PP-002
Appendix I	Enhancement	Added criteria for CT RCP

Revision: 13	**Effective Date:** Jul 24, 2015	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Scope	Reflects current LOQs and target compounds	Updated to the current LOQs and added Chloramben
Reagents and Standards	Reflects current standards	Updated Picloram in the Herbicide intermediate. Updated hexachlorophene in the ICV intermediate Updated Prep scheme in table.
Quality Assurance/Quality Control	Enhancement	Added information on evaluating the internal standard.

Reference

1. *Test Methods for Evaluating Solid Waste*, SW-846 Method 8151A, December 1996.

2. State of Connecticut Department of Environmental Protection, Recommended Reasonable Confidence Protocols for Chlorinated Herbicides by SW-846 8151, version 2.0 July 2006.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0816, 11110, 11111	Extraction of Chlorinated Herbicides in a Water Matrix by SW-846 8151A
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015493	QC Data Acceptability and Corrective Action
1-P-QM-PRO-9015494	Interpretation of Chromatographic Data
1-P-QM-PRO-9015496	Monitoring of QC Data Acceptance Limits
1-P-QM-PRO-9015498	Setting Up Single Component Initial Calibrations
1-P-QM-PRO-9015501	Common Equations Used During Chromatographic Analyses

Scope

This method is applicable to the measurement of the following chlorinated herbicides in water and wastewater:

Compound	Limit of Quantitation (µg/L)	Notes
2,4,5-T	0.05	
2,4,5-TP (Silvex)	0.05	
2,4-D	0.5	
2,4-DB	1.0	
2,4-DP (dichlorprop)	0.5	
Dalapon	1.25	
Dicamba	0.3	
Dinoseb	0.5	
Hexachlorophene	0.2	Special request required
MCPA	200	
MCPP (mecoprop)	200	
PCP	0.05	
Picloram	1.0	Special request required

LOQs are based on annual statistical evaluation of laboratory data and are subject to change. The current LOQs are maintained in the LIMS.

See Table I for a list of the compounds in each scan. See 1-P-QM-WI -9015078 (Analysis #0816, 11110, 11111) for the extraction procedure.

Basic Principles

Samples are hydrolyzed to convert methyl esters to the acid state. Extraneous material is removed using a methylene chloride solvent wash. The chlorinated phenoxy acids are extracted from an acidified sample of water or wastewater with ethyl ether. Acids are then converted to their methyl esters using diazomethane as the derivatizing agent. The methyl esters are determined by gas chromatography using an electron capture detector (GC-ECD). A florisil cleanup and/or dilution can be performed to eliminate matrix interferences that introduce large unresolvable peaks in the chromatogram. A dilution is required if interferences such as chlorinated acids and phenols are present. Refer to 1-P-QM-PRO-9015477 for more details on this cleanup procedure.

Interferences

An electron capture detector is very sensitive to compounds that contain halogens and also responds to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur. Plastic must not be used during the extraction or analysis to prevent phthalate contamination. Glassware must be scrupulously cleaned.

This analysis can be particularly affected by matrix interferences or coextractives that include chlorinated hydrocarbons, phthalates, organic acids, and phenols. A florisil cleanup is used to reduce these types of interferents. The extraction procedure also includes a cleanup using methylene chloride after alkaline hydrolysis of the analytes of interest.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards and handling sample extracts and solvents. Lab coats and glasses are required while working in the laboratory areas.

All GC vials and extract vials are placed in a hazardous waste container for lab pack disposal. There is a satellite container in the laboratory that is then emptied into the main laboratory waste collection drums. All solvent waste is disposed of in designated solvent waste containers in the laboratory which are then combined with the main laboratory designated waste collection drum.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass bottles and preserved with sodium thiosulfate. Samples must be kept cool at 0° to 6°C, not frozen. They must be extracted within 7 days of the date collected, and extracts must be analyzed within 40 days.

Apparatus and Equipment

1. HP7890 gas chromatograph fitted with electron capture detector or equivalent
2. Columns:
   a. Phenomenex ZB-35 30 m × 0.32 mm × 0.5 µm
   b. Phenomenex ZB-XLB 30 m × 0.32 mm × 0.25 µm
3. Integrating system such as Chrom Perfect by Justice Innovations or equivalent
4. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents:

1. Hexane, pesticide grade for autosampler rinse vials. Stored at room temperature.

2. UPC (ultra pure carrier) nitrogen for detector make-up

3. UPC helium for carrier gas

4. UPC hydrogen carrier, bottled or from a generator

5. Ethyl ether, high purity, nonpreserved – Store at room temperature.

B. Standards:

1. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

2. All standards are prepared using Class-A volumetric flasks, pipettes, and syringes.

3. All standards are stored in labeled vials or flasks in a freezer at $\leq$-10°C.

4. DBOFB stock – 4,4'-dibromooctafluorobiphenyl Ultra PPS-170 at 1000 µg/mL ppb, or equivalent. This is used as the internal standard.

5. DBOFB Intermediate – Dilute 1.0 mL of DBOFB stock into 50 mL of acetone. This solution is stable for 6 months.

6. Working DBOFB IS diluting solution – Dilute 5.0 mL of the DBOFB intermediate into 100 mL of hexane. This solution is used to make sample dilutions. This solution is stable for 6 months.

7. DCAA SS stock – 2,4 dichlorophenylacetic acid (DCAA) non-methylated stock. Ultra PPS-162 at 5,000 µg/mL, or equivalent.

8. Acid herb stock – Acid herbicide stock for calibration standards: Accustandard catalog #S-12214-R5 in methanol (concentrations vary per compound), or equivalent.

9. Hexachlorophene stock – Restek cat #31811 at 2,000 µg/ml in methylene chloride, or equivalent.

10. Picloram stock – Restek cat #32265 at 1,000 µg/ml in methanol, or equivalent.

11. Herbicide intermediate – Dilute 1.0 mL of acid herbicide stock, 0.02 mL of hexachlorophene stock, 0.05 mL of picloram stock, and 0.02 mL of DCAA SS stock into 10 mL of ethyl ether. This solution is methylated following procedure outlined in Analysis #4181 prior to making the calibration standards. This solution is stable for 6 months.

12. MS Herbicide stock:

a. Herbicide mix in the acid form for the matrix spiking solution: Ultra cat #HBM-8150A in methanol (concentrations vary per compound), or equivalent.

b. PCP stock – Accustandard cat #APP-9-176-D-20X at 2000 µg/mL in methylene chloride, or equivalent.

c. Dinoseb fortification stock – Restek cat #32251 at 1,000 µg/ml in methanol, or equivalent.

d. Picloram stock – Restek cat #32265 at 1,000 µg/ml in methanol, or equivalent.

e. Hexachlorophene stock – Restek cat #EPA-1125 at 1,000 µg/ml in acetone, or equivalent.

13. MS MCPA stock – Neat compound from ChemService cat #PS-40, or equivalent. Dilute approximately 0.25g into 5 mL of methanol. Stable for 1 year.

14. ICV Stocks:

a. Acid herbicide mix – Supelco cat # 46861-U in methanol (concentrations vary per compound), or equivalent.

b. Hexachlorophene – Accustandard cat #APP-9-116 at 100 µg/l in methanol, or equivalent.

15. ICV Intermediate – Dilute 1 mL of acid herbicide stock, 0.5 mL of hexachlorophene stock, and 0.02 mL of DCAA SS stock into 10 mL of ethyl ether. This solution is methylated following procedure outlined in Analysis #4181 prior to making the working standard.

16. Surrogate Stock (SS) – Ultra Scientific cat #PPS-161 containing 2,4-Dichlorophenylacetic acid (DCAA) in MTBE, or equivalent.

17. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. HERB 1), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 40 fold of the initial calibration level.

18. Prepare the spiking solutions using the prep scheme in the table below:

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description	Expiration I
MS (herb)	Chlorinated Herb Stock	2.5	100	Methanol	Herb Spike	6 month
	PCP Stock	0.05				
	Dinoseb Fortification Stock	0.3				
	Picloram Stock	0.25				
	Hexachlorophene Stock	0.12				
MS (MCPA)	MCPA Stock	0.5	100	Acetone	MCPA fortification mix	6 month
IS	4,4 DBOFB Stock solution	1	50	Hexane	Internal Std	6 month
SS	SS DCAA Stock	1.0	1000	Hexane	Herb surrogate standard	
IBLK	SS (DCAA methyl ester)	0.80	200	Hexane	Instrument Blank	

GC Conditions

The conditions listed are optimum however, they can be changed to improve the linearity, sensitivity, or overall chromatography on each GC system as needed.

Instrument setup (primary and confirmation)

Detector – ECD
Detector temperature – 300°C
Carrier – Hydrogen at 10 psi (Helium is a substitute)
Makeup gas – N_2 at 30 mL/min for Varian ECDs, 55 mL/min for HP ECDs
Injection size – 2-µL, direct injection
Injector temperature – 250°C
Oven temperature – 50°C, hold 0.5 min, 25°C/min to 100°C, 12C/min to 310°C Hold 2 min
Carrier – Hydrogen at 12 PSI (helium is a substitute)

Calibration

1. Prior to starting a new calibration, fill the autosampler rinse vials with clean solvent or replace vials themselves if they appear to be dirty.

2. Prepare a sequence as follows:

1. Conditioner
2. IBLK
3. Herb Level 1
4. Herb Level 2
5. Herb Level 3
6. Herb Level 4
7. Herb Level 5
8. Herb level 6 (optional)
9. MDHEX
10. ICHBX
11. Blank
12. LCS
13. 1234567
14. 1234567ms
15. 1234567msd

16. – 20. Continue with samples

21. Herb Level 3

22. – 31. Ten samples

32. Herb Level 3

Continue running groups of 10 samples followed by an Herb level 3 standard between sample groups.

3. The conditioner injection is usually a standard or sample that has already been injected.

a. Any number of conditioners can be used prior to the start of the run – one is shown here as an example.

b. It is used to prime the system and is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

c. Hexane blanks are run to allow the GC to go through some temperature program runs and/or to check the cleanliness of the system.

4. The instrument blank (IBLK) is injected after the conditioners but before the initial calibration.

a. It is used to determine that the instrument is free of background noise or contamination.

b. IBLK may also be run with the continuing calibration standards – this is optional, but is frequently requested for projects.

5. Initial Calibration (ICAL)

a. The system is calibrated using a minimum of five concentration levels.

b. An internal standard calibration is used with average response factor (AVGRF) for all analytes where the %RSD is ≤20%.

(1) If the average of the %RSDs of all compounds in the initial calibration standard is ≤20%, the AVGRF is used for all compounds in the initial calibration when needed.

(2) Alternatively, when these criteria are not met, a calibration curve must be used.

c. When using a calibration curve, a linear fit must be tried first.

(1) Use the linear fit if the correlation coefficient is >0.99.

(2) However, if the correlation coefficient is <0.99, a quadratic fit must be tried. A 6−point calibration must be run to use quadratic.

d. For either curve type, extrapolate or force zero is not allowed.

e. See 1−P−QM−PRO−9015498 for details on using Chrom Perfect for setting up single component calibration files.

f. Ensure all peaks in the standards are labeled properly and that the scaling is such that concentrations at the MDL exhibit a peak about 2 to 3 mm in height.

g. Be sure all peaks in the MDLH standard are integrated by the data system.

6. Initial Calibration Verification (ICV)

a. Verification of the calibration curves is performed using the ICV mixtures injected directly after the full ICAL.

b. The % difference of the concentrations for each analyte must be within ±15% difference of the nominal concentration for the curve to be used for sample analysis.

7. Continuing Calibration Verification (CCV)

a. A CCV standard is analyzed after every ten injections (samples, QC, blanks etc) using the level 3 calibration standard.

b. The CCV between samples must exhibit a response at ±15% difference (%D) for each compound, or the average of the %Ds must be within ±15% for that standard to be compliant on at least one of the two columns used for the analysis.

c. The concentration calculated for the continuing injection is compared to the nominal concentration.

d. Samples must be bracketed with compliant standards.

(1) Exception: If, however, the standard following a sample is outside the ±15% but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected.

(2) If confirmation of target analytes is needed, then the second column should meet the 15% continuing calibration criteria, as well as all initial calibration criteria.

8. If an instrument blank (IBLK) is injected after the CCV, it must be evaluated as a water matrix against the water MDL/LOQs.

a. The IBLK must not have any target compounds above the reporting limits.

b. If a target analyte is detected in the IBLK, any associated samples with detection for that same target must be evaluated.

(1) Unless the concentration in the sample is more than 10× the IBLK value, the sample must be reinjected after another compliant IBLK

(2) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up the instrument.

9. Retention Time (RT)

a. RT windows are established as 3× the standard deviation determined over a 72-hour period, or at no less than ±0.03 min, applied to the midpoint initial calibration standard.

b. If the RTs for a CCV fall outside the windows, update the midpoint RT using that standard.

(1) Save this under an appropriate name to indicate an update has occurred.

(2) All subsequent continuing standards run within a 24-hour period must fall within this window.

(3) Retention times cannot be updated more than once per day.

(4) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Retention times of peaks in the samples are compared to the standard RT windows.

a. Peaks that are present on both columns are quantitated and the high value is reported unless there are chromatographic anomalies.

b. See 1-P-QM-PRO-9015494 for more information on the interpretation of chromatographic data.

2. Samples that contain levels of analytes above the highest level calibration standards must be diluted and reanalyzed. Use Working DBOFB IS diluting solution to prepare dilutions. See standard section.

When preparing dilutions, add sufficient internal standard to maintain the same 100-µg/L concentration.

3. If data is reported using the grand mean for the ICAL or CCV, the following comment(s) will be added to the report:

a. 1571 - The % difference for the calibration verification standard is outside the ± criteria for the analyte(s) listed below. Since the average of the % difference values meets the criteria, the results are reported. This applies to the following analyte(s):

b. 1572 – The % relative standard Deviation for the initial calibration for the analyte(s) listed below is above the 20% criteria. Since the average of the RSD values for all calibrated compounds meets the criteria, the average response factor calculation was used. This applies to the following analyte(s):

c. **NOTE:** Use of the average of the %Ds (grand mean) for continuing calibration standards is not permitted for samples from **South Carolina** and may not be approved for specific client projects.

Calculations

1. Sample Concentration

$$Sample\ Concentration,\ \mu g/L = Extract\ Conc\ (\mu g/L) \times \frac{DF \times FV}{IV} \times Conc\ Int\ Std$$

Where:

IV = Initial volume in mL

DF = Dilution factor

FV = Final volume in mL

2. Linear Curve

$$Extract\ Conc\ (\mu g/L) = \frac{(pk\ ht\ /\ int\ std\ ht) - Y - intercept}{slope}$$

3. Average response factor (AVGRF)

The calculation is the same as above except the extract concentration is calculated as follows:

$$\text{Extract Conc.}\,(\mu g/L) = \frac{\text{pk ht in sample}}{\text{internal pk ht (of std) in sample}} \;/\; \text{AVG RF}$$

Where:

$$\text{AVG RF} = \frac{\text{RF Calib 1} + \text{RF Calib 2} + \ldots + \text{RF Calib 5}}{5}$$

$$\text{RF} = \frac{\text{Standard Peak Height / Internal Peak Height}}{\text{Standard Concentration}\,(\mu g/L) \,/\, \text{Conc Internal Std}}$$

4. Usually, the internal standard concentration is normalized to 1. It can be changed for injections that have a concentration different than the normal due to dilution or alternate amount added during extraction.

5. See 1-P-QM-PRO-9015501 for more details on the calculations related to the calibration.

Statistical Information/Method Performance

MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. The full study is initially run on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Results from all instruments are compared and pooled together to determine the reporting MDL. NELAC allows for an annual verification of the MDL in lieu of an EPA MDL study. Copies of the annual studies are maintained by the department supervisor. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department Database is updated via a download from the LIMS. QC Acceptance limits are established as statistical limits. See 1-P-QM-PRO-9015496 for further information on monitoring and establishing limits

Quality Assurance/Quality Control

At least one blank and LCS is analyzed with each batch of 20 samples. An MS andMSD is analyzed with each batch of 20 samples as long as there is ample volume. An LCSD is performed if an MS/MSD

cannot be done. The spiking solutions contain all analytes of interest. A surrogate standard of 2,4−dichlorophenyl acetic acid (DCAA) is added to each sample, blank, and spike to monitor the efficiency of the extraction and the operation of the autoinjector. An internal standard is added to each sample, blank, and spike. The internal standard (DBOFB) response for each sample and QC is evaluated by comparing the response in the sample to the response of the nearest check standard. Since there is no criteria listed in the method, an advisory window of 50 -150 is used.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed instead.

1−P−QM−PRO−9015493 outlines the QC acceptability and corrective action.

Table I

Compound	0952 (TCLP)	10407 master
2,4-D	X	X
2,4,5-TP (Silvex)	X	X
2,4,5-T		X
Dinoseb		X
Dalapon		X
MCPP		X
MCPA		X
2,4-DP		X
2,4-DB		X
PCP		X
Hexachlorophene *		X
Dicamba		X
Picloram *		X

* Denotes Special Permission required to add these compounds since they are not routine analytes.

Appendix I

CT RCP Requirements

No use of grand mean for initial and continuing calibration evaluation.

If any sample needs a comment about out of spec data, comment 2510 must be put on the sample first, followed by our comment, so that the RCP form generates properly from the LIMS.

Comment about any raised limits (does not need precursor comment)

Need instrument blanks after every CCV set.

If there is >40% difference between column A and B, a comment must be placed on the analytical report. There are two comments for this: 1570 says the higher result was reported, 1569 says the lower result was reported. Use these on the analytical report as they apply.

The LCS recoveries must be between 40 -140%.

The MS/MSD recoveries must be between 40-140% with the RPD ≤ 30%.

The surrogate recovery must fall between 30 – 150%.

End of document

Version history

Version	Approval	Revision information
14	26.AUG.2016	

eurofins	Always check on-line for validity **Metals by ICP for Methods SW-846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)**	Level: **Work Instruction**
Document number: **T-MET-WI11931**		
Old Reference: **1-P-QM-WI-9018442**		
Version: **10**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **30-JUN-2017**	Document users: **6_EUUSLA_Metals_ICP Analysis, 6_EUUSLA_Metals_ICP Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

Table of Contents

Revision Log

Revision: 10		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Enhancement	Added method 6010D
Throughout Document	Reflects re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Throughout Document	New version of the method that is being supported	Added 6010D requirements
Purpose	Unnecessary section. Information contained within the scope	Removed section
Definitions	Contained within higher level documents or within this document	Removed section
Personnel Training and Qualifications	Higher level documents not required to be referenced	Removed reference to document 1-P-QM-QMA-9015390 and 1-P-QM-QMA-9017325.
Sample Collection, Preservation, and Handling	Change in process	Removed that sample storage is the required personnel to check the pH of the water samples. This requirement can be performed by the metals department.

Revision: 10	**Effective Date:** This version	
Routine Maintenance for the ICAP 6000 Duo Analyzer	All information is contained within a higher level document.	Removed documentation for instrument/analysis tag out and return to service.
Quality Assurance/ Quality Control E.12.	Contained within higher level document	Removed MCL values
Quality Assurance/ Quality Control G.	Addition	Added IEC frequency information
Table II	Enhancement	Added 200.7 SIC requirements for Wisconsin

Revision: 9	**Effective Date:** Nov 20, 2015	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Enhancement	Added the acronym ICP
Throughout Document	Reflects change in naming convention	Changed Parallax to LIMS.
Definitions	Enhancement	Included Linear Range criteria and frequency, and IECs.
Personnel Training and Qualifications	Enhancement	Added additional information for DOCs.
Sample Collection, Preservation and Handling	Clarification Correction	Clarified that the holding time of 180 days includes the analysis. Changed from pH >2 to pH ≥2
Statistical Information/ Method Performance	Information is already discussed in an earlier section	Removed all information concerning IDOCs and training documents
Instrument Operations A.5.	Addition	Added IEC requirements specific to WI.
Table I and Table II	Correction	Updated Linear Range criteria and frequency.
Table II	Clarification	Noted that the LCS is spiked at or below the MCL for all primary drinking water metals. Added new EW rule for the PB and LCS if they are out of specification data cannot be accepted for any reason. Clarified PB requirements.

Reference

1. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010B, December 1996.

2. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010C, February 2007.

3. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010D, Rev.4, July 2014.

4. Method 200.7 (rev. 4.4), Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, USEPA 600/R-94/111 May 1994.

5. ICAP™ 6000 Series ICP-OES Spectrometer Operator Manual, 2005/2006.

6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-MET-FRM8802	ICP LOQs (mg/L)
T-MET-FRM9070	MSA Prep for Samples with MS/MSD <50%
T-MET-FRM9072	MSA Prep for Samples within TCLP Limits
T-MET-WI12063	Working Instructions for Preparation of ICP Solutions and Standards
T-MET-WI9082	Working Instructions for Prep Solutions and Standards
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope

This procedure applies to analyses performed in the Metals department using Inductively Coupled Plasma (ICP) Atomic Emissions Spectroscopy for identification and quantitation of metallic constituents by Methods 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous).

The limits of quantitation (LOQ) are based on annual statistical evaluation of laboratory data and are subject to change without notification. The current method detection limits (MDLs) and LOQs are maintained in the laboratory information management system (LIMS).

This SOP also outlines the proper operation and maintenance of the ICP instrumentation and provides consistent guidelines for the evaluation of ICP data.

Routine Methods

Elements routinely analyzed on the Thermo Scientific iCAP 6000 Series Analyzer include LIMS analyses #:

Element	Waters Analysis #	Solids Analysis #	Wavelength (nm)
Ag	7066	6966	328.06
Al	1743	1643	308.21
As	7035	6935	189.04
Au	11762	11761	242.80
B	8014	7914	249.67
Ba	7046	6946	455.40
Be	7047	6947	313.04
Ca	1750	1650	317.93
Cd	7049	6949	226.50

Co	7052	6952	228.62
Cr	7051	6951	267.72
Cu	7053	6953	327.40
Fe	1754	1654	261.19
K	1762	1662	766.49
Li	1756	1656	670.78
Mg	1757	1657	285.21
Mn	7058	6958	257.61
Mo	7060	6960	202.03
Na	1767	1667	589.59
Ni	7061	6961	231.60
P	10143	10145	177.49
Pb	7055	6955	220.35
S	12004	12003	182.03
Sb	7044	6944	206.83
Se	7036	6936	196.09
Si	1765	12763	251.60
Sn	7069	6969	189.99
Sr	8068	7968	421.55
Te	13494	13498	214.281
Th	13495	13499	401.913
Ti	7070	6970	334.94
Tl	7022	6925	190.86
V	7071	6971	292.40
W	13496	13500	207.911
Zn	7072	6972	213.86
Zr	10144	10146	339.19

Basic Principles

An instrument run sheet is prepared using the information provided on the sample batch sheet received from the prep area. Appropriate standards, check standards and interference check standards are added. Standards and samples are poured as needed to be analyzed on the instrument.

Samples are received from the prep area in Nalgene containers. The samples are analyzed directly on the instrument, with the exception of the spiked sample and a serial dilution of the same background sample. The spiked and serial dilution sample is prepared in a volumetric flask or directly into the graduated plastic digestion tube that is placed on the autosampler for instrument analysis. Any sample that requires a dilution is prepared in the same fashion. Standards and samples are entered in to a sequence file on the instrument in the same order as on the ICP run sheet.

Water and soil samples are treated with acids and heated to solubilize the metals present. These digestates are then analyzed for trace metals by an atomic emission spectroscopic technique. Samples are transported to a nebulizer via an autosampler and peristaltic pump. The nebulizer introduces an aerosol into a spray chamber; the resulting mist is then transported to an argon plasma torch where excitation of atoms occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency (R.F.) inductively coupled plasma. The spectra are dispersed by a diffraction grating and the

intensities of the light at each wavelength are monitored by a photosensitive device. The signals from the photosensitive device are processed by a computer. A background correction technique is required to compensate for variable background contribution to the spectra of trace elements.

Interferences

Spectral interferences are caused by background emission, stray light from high concentration elements or overlap from a spectral line from another element. Spectral interferences are compensated for by the use of background points, alternate wavelengths and interelement corrections.

Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for by using internal standardization.

Memory interference, or carryover, is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time of the autosampler tubing overcomes any memory interference.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO_3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood.

Hydrogen Peroxide 30% (H_2O_2) - This oxidizer can cause skin burns. Always use in a fume hood.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the sequence editor to set up the run, perform calculations, interpret raw data, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an IDOC (Initial Demonstration of Capability).

The IDOC consists of four laboratory control samples (LCS) that are carried through all steps of the prep and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Sample Collection, Preservation, and Handling

A. Aqueous samples are collected in plastic or glass containers and are preserved with nitric acid. Solid samples are collected in glass containers with no chemical preservation. All samples are typically shipped and stored at 0°to 6°C, not frozen, but room temperature is also acceptable. All samples must be digested and analyzed within 180 days of collection. Sample digestates are stored in plastic bottles at room temperature.

B. pH Adjustment

1. Upon receipt at the laboratory, personnel check the pH of water samples. If the pH is ≥2, the pH of the sample is adjusted to a pH less than 2 with nitric acid. The date and time that the additional preservation was added is recorded. After a minimum of 24 hours, prior to digestion, the Metals department checks the pH of the sample to confirm that the pH is less than 2.

2. Drinking water samples require pH check immediately before digestion. If the pH is ≥2, the pH of the sample is adjusted to a pH less than 2 with nitric acid. The date and time that the additional preservation was added is recorded. After a minimum of 24 hours, prior to digestion, the Metals department checks the pH of the sample to confirm that the pH is less than 2.

3. Dissolved Metals - Samples to be analyzed for metals requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt. Samples are filtered into containers and preserved to a pH of <2 with HNO_3.

C. Sample Discard - The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and verified in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

The following is a list of the hardware and apparatus necessary for ICP analysis. More detailed hardware information is located in the *Operator's Manuals*.

1. 15−mL graduated polypropylene screw cap tubes (certified ±1%)

2. 16−mL polystyrene tubes

3. Filter paper – Whatman No. 540, 90−mm ashless

4. FilterMate filtration device with 0.45−µm PTFE fiber filter and insertion tool

5. 1 × 100 10−mL sterile disposable syringes

6. 25-mm syringe filters, PTFE, 0.45−µm

7. 30-mL polypropylene medicine cups

8. Calibrated electronic hand−held pipettes and tips (10 – 5000 µL) - FisherBrand or equivalent.

9. Spectrometer - The Thermo Scientific iCAP™ 6000 series Trace Analyzer utilizes a high performance solid state Charge Injection Device (CID) camera system to deliver high contrast/low noise imaging and quantification of all wavelengths in the analytical range. With the entire spectrometer and foreoptics purged with either Argon or Nitrogen, it features a 52.91 grooves/mm grating and dual-view detector.

10. Auto-sampler – The ESI SC-14 auto–sampler and integrated “FAST” system offer increased capacity and reduce sample introduction times. The parameters for each automated run are entered into the auto-sampler table in the iTEVA™ software as described in Instrument Operation, Section A of this SOP.

11. Peristaltic pump – The peristaltic pump regulates the flow of the following: sample, internal standard, instrument rinse and spray chamber waste. Special care must be taken to ensure that all pump tubing is connected properly. The Teflon concentric and glass V-groove nebulizers have a natural uptake, but a peristaltic pump is used to compensate for differences in sample viscosity. After traveling through the peristaltic pump, the sample and internal standard tubing are combined by a “Y” connector, and then allowed to mix in a mixing coil before entering the nebulizer.

12. R.F. generator –The iCAP™ 6000 series Trace Analyzer utilizes an internal solid state RF Generator at 27.12MHz with a power efficiency greater than 78%.

13. Coolflow –The ThermoFlex™ 900 cooling device for the iCAP™ 6000 series Trace Analyzer operates at 17°C.

14. Personal computer – The iCAP™ 6000 series Trace Analyzer is controlled by PCs.

Reagents and Standards

Reagent and standard information and the preparation of the following standard and solutions are located in T-MET-WI12063:

- Initial Calibration (ICAL)
- Blanks and calibration standard
- Initial Calibration Verification (ICV) standard
- Continuing Calibration Verification (CCV) standard
- Rinse/Carrier and Profile Solutions
- Interference Check Solutions (ICSA and ICSAB)
- Low Level Check (LLC) standards
- Post Digestion Spike (PDS)
- Linear Range Standards (LRS)
- Internal Standard Solution

Instrument Detection Limit (IDL) and MDL Solutions
Matrix Matched Standards

Calibration
See Tables I and II for the frequency, acceptance criteria and corrective action of the ICAL, ICV, ICB, LLC, ICSA/ICSAB.

Procedure
A. Setting up an ICP run

1. Determine the batches to be analyzed and determine any special requirements by viewing lab notes and/or project notes that are with the batch paperwork delivered from the metals prep area.

2. Assign an ICP metals storage location and document the location on the batch sheet(s) and on the sample lid for the Prep Blank of each batch.

3. Log in to LIMS and select Sequence Editor from the IDAT menu. In the Sequence Editor window, select the appropriate batch digest type (EPA or SW-846).

4. Choose the appropriate digest from the list, and click on “Get Batches”

5. Select a batch from the list to display the incomplete samples in that batch.

6. Select the appropriate template (pre-designed with the correct QC standards and auto-sampler locations) from the template list; a blank form is opened. If that batch has been previously documented, the existing sequence file is also loaded.

7. Add all required samples into the form, either by choosing “Add batch” for all samples, or by “dragging” individual samples into the field.

a. Sample names include:

PBW – Prep blank (water)
LCSW – Laboratory control sample (water)
LCSDW – Laboratory control sample duplicate (water)
PBS – Prep blank (solid)
LCSS – Laboratory control sample (solid)
LCSDS – Laboratory control sample duplicate (solid)

ELLEs' sample number
CCV – Continuing calibration verification
CCB – Continuing calibration blank
LLC – Low level check
ICSA – Interelement correction standard – A
ICSAB – Interelement correction standard – AB

b. Batches with only field blanks or equipment blanks do not need a post-digest spike or a serial dilution.

c. "As Received" samples must be run with a blank and LCS, LCSD (prepared and documented in LLENS by the analyst).

8. Edit dilution factors (DF), protocols, and auto-sampler locations as needed. Consult the Incomplete List sheet to determine the analysis requirements for each sample.

9. Remove all unnecessary QC standards by clicking on the "QC" button. Unnecessary lines are removed by selecting individual lines and clicking on the red "X" button.

10. Save the sequence file. Click "OK" to acknowledge that the file has been saved and the cover sheet document has been created. The document opens automatically.

11. Edit the cover sheet document as needed, including batch location information and any additional comments or instructions.

12. When setting up a run, Batch QC must be placed in the same block of ten or fewer samples. If there are two LCSs, they must be placed one after the other. [The order of the batch QC is typically PB, LCS, (LCSD), Bkg, PDS, DUP, MS, (MSD) and SD.]

a. ICV/ICB must be analyzed immediately after the calibration curve.

b. CCV/CCB must be analyzed after every ten analytical samples.

c. LLC, ICSA, ICSAB, CCV, CCB must immediately follow the ICV/ICB and must conclude each run. (**NOTE:** As of 10/11/2012 for all DOD protocols, it is not necessary to analyze the ending ICSA/ICSAB and LLC check samples. This is for DOD only!)

d. Any deviations from protocol must be noted in the Comments Section of the cover page

e. Any unused portion of the run sheet must be "Z'd" out with initial and date.

B. Pouring an ICP run

It is important to minimize any chance of contamination, both of yourself and the samples. Keep your hands and the work area clean at all times. Do not re−use any pipette tips.

Run QC standards are prepared separately, and kept in separate auto-sampler racks to be obtained at the time of analysis.

1. Use the batch location information on the selected run sheet to locate the corresponding batch(es). In the “Poured by:” section of the header, record: initials, employee number, and the date.

2. Carefully examine the batch to ensure there are no discrepancies between the Batch Preparation Sheet, run cover sheet, and the physical placement of samples in the batch.

3. Obtain and label all required test tubes, and place them in test tube racks. Any poured sample that does not require a graduated test tube is to be poured into a polystyrene tube.

4. Prepare and label the PDS required for each new batch (sample volume permitting).

a. A PDS is prepared using 0.2 mL of a custom-ordered PDS solution into 9.8 mL of background sample.

b. Equivalent amounts of the custom PDS must be used if decreasing sample volume.

c. Record preparation details in the comments column of the ICP run sheet.

5. Prepare a serial dilution by diluting the background sample by 5×.

a. If the background sample chosen for serial dilution has been diluted due to matrix interference or to bring the concentration into the linear range of the instrument, the serial dilution must also be diluted by 5 times the dilution factor of the background (i.e., if Bkg = DF5, S.D. must = DF25).

b. Document preparation details in the comment section.

6. Using Whitman No. 540 filter paper or the Filter Mate filtration device, filter those samples that are cloudy or contain particulate.

a. If the filtrate remains cloudy, filter again.

b. Samples with limited sample volume must be filtered using a 10−mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter.

c. If any samples are filtered, the prep blank must also be filtered.

d. Document all filtrations on the run cover sheet and mark each sample lid with an “F” to indicate that it was filtered.

7. For TCLP samples that requires method of standard additions, refer to T-MET-FRM9072 or T-MET-FRM9070 for instructions.

8. Perform any additional spiking or dilutions and document preparation details in the comment section.

9. Cover the samples with lids or with plastic wrap to prevent contamination.

10. Place the poured batch on the bench top to await analysis, or return samples to their ICP sample storage location.

11. Take **NOTE** of the following:

a. A post−digest spike and a serial dilution must be performed on one sample in each digestion batch. Typically, the background sample is chosen. If the batch QC is split between two samples, the post-digest spike is performed on the background sample accompanied by a matrix spike; the serial dilution is performed on the background sample accompanied by a matrix duplicate. If sample volume is limited, it is acceptable to use the duplicate for the PDS and SD.

b. Analysis information, including standard lot numbers, run number, rinse time, method, analyst and date of analysis, are documented on the cover sheet at the time of analysis.

c. Documentation is of utmost importance. Double check all entries.

d. Dilute samples when necessary to yield a response that falls within the calibration range. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

e. If a batch requires re-analysis, it is acceptable to re-use dilutions and/or spiked samples that were prepared for the previous analysis. A “P” in the comments section is used to indicate that a previously poured test tube is being re−used.

Instrument Operations

A. iTEVA™ software: The Thermo Scientific iCAP™ 6000 series instrument is operated through iTEVA™ software. From the Start menu, select “iTEVA™ Control Center” to start the software.

1. Plasma ignition of the iCAP™ 6000

a. Open the plasma status window by clicking on the icon at the bottom of the screen.

b. Verify that all parameters are within acceptable range for ignition as indicated by a green light.

c. Ensure that the drain tubing for the spray chamber is properly connected to the peristaltic pump and positioned to drain into a waste carboy.

d. Select “Ignite plasma”. After the plasma has ignited, the instrument automatically performs optimization of the nebulizer gas pressure, and then starts the on-board peristaltic pump.

e. If the plasma does not light, repeat steps b-d.

f. Once the plasma operating parameters have engaged, exit the Plasma status window by clicking on Close.

g. If the plasma has been off for more than 15 minutes let the instrument warm up for 30 minutes. If the plasma has been off less than 15 minutes, let the instrument warm up 5 to 10 minutes.

2. Automated analysis using the iTEVA™ software

a. Open the Analyst window from the Control Center by clicking on the Analyst icon. When prompted, select the appropriate method.

b. In the Analyst window, click on the Sequence tab at the bottom of the screen.

c. From the Auto-session menu, select Open new session.

d. The “New auto-session” window opens. Choose the appropriate autosampler configuration set.

e. Click on “New” to add a sequence and the “Add sequence” window appears.

f. Click on the “Import comma delimited text file” option and choose an appropriate template or previously prepared run sequence from the drop-down menu for the run to be entered. Click OK to close this window and continue.

g. Change the sequence name to reflect the run number, but do not change the method revision number.

h. Click OK at the bottom of the “New auto-session” window to continue.

i. The sequence template is loaded. When prompted, click OK to choose the “Use positions” option, then Yes to accept duplicate positions.

j. Edit the table as needed to include sample number, class, dilution factor, batch number and protocol for each sample.

k. Additional samples and QC are added using the “Add sample” and “Add QC” icons, respectively, at the top of the screen. Unused rows are removed by selecting the entire row(s) and using the “remove sample” icon at the top of the screen.

l. Right-click on the calibration standards list and select “Auto-locate” to have autosampler positions automatically assigned.

m. Verify that the sample list begins and ends at the correct tube numbers, and check all entries for errors.

n. Click on the “Initialize Autosampler” icon to initialize the autosampler for the current configuration.

o. Click on the printer icon and choose to print page 1 only. This printout is kept with the run cover sheet.

p. Carefully examine the batch to ensure there are no discrepancies between the run cover sheet, auto-sampler sequence table and the physical placement of samples in the batch.

q. Click on the “Start automated run” icon to start the run sequence. If a run sequence is to be started at a sample or standard other than the initial calibration, right-click on the appropriate sample and choose “Start Auto-Session Run at this Sample” and elect not to run the “start actions.”

3. Running an AutoPeak in iTEVA™

a. In an open autosampler session (autosampler must be initialized), right-click on the location of the AutoPeak solution, and choose “Go to...”

b. Click on the Analysis tab to open the Analysis window.

c. From the Instrument menu, select Perform AutoPeak.

d. Click on All elements, then OK.

e. When prompted to aspirate the high standard, click OK. The AutoPeak is now performed.

f. When the AutoPeak has finished, click on Done.

4. Manual analysis in iTEVA™

a. In an open autosampler session (autosampler must be initialized), add sample(s) to be analyzed to a new or existing sequence as detailed in section A.2 of this SOP.

b. To start analysis, right-click on the appropriate sample and choose “Start Auto-Session Run at this Sample” and elect not to run the “start actions.”

c. When analysis is complete, results can be printed from the Analysis tab by right-clicking on the sample name and selecting “Print Sample…”

5. Performing an interelement correction (IEC) in iTEVA™

a. Prepare a solution of the interfering element at the linear range of the instrument. Perform manual analysis according to section A.4 of this SOP.

b. Note results of elements with a known interference that are greater than the limit of quantitation.

c. Divide the result of each interfered element by that of the interfering element. These values represent the amount the current IECs need to be adjusted.

d. In the Method window, click on Elements to expand the list of elements in the method.

e. Click on the targeted element to view the settings for that element.

f. Click on the IECs tab to view the current IECs for that element.

g. Add each calculated result to the correction factors currently in the first column corresponding to each pair of elements.

h. Enter the new correction factors in the table.

i. Repeat steps d-f as needed.

j. Click on the Save method icon to save the method.

k. For Wisconsin samples analyzed by EPA 200.7 run the following IEC check.

(1) For interferences from iron and aluminum, only correction factors (positive or negative), when multiplied by 100, that exceed ± LOQ need be tested on a daily basis.

(2) For the all other interfering elements, only those correction factors (positive or negative), when multiplied by 10, that exceed the ± LOQ need be tested on a daily basis.

(3) If the correction routine is operating properly, all interferences should fall within ± LOQ. **NOTE:** When making an update to an IEC, the interference should be analyzed a second time to confirm that the IEC is correct.

(4) If the correction factors tested on a daily basis are found to be within ± LOQ for five consecutive days, the verification frequency may be extended to weekly. **NOTE:** If the samples do not contain concentrations of the interfering elements greater than 10 ppm, daily verification is not required.

B. Import and QC review of run data

1. Open the iCAP Data Reprocessor and ensure the correct database is listed.

2. Click on “Get run name list”. Select the appropriate run. Click on “Reprocess”.

3. Start the LIMS software and log in. Select Import from the IDAT menu.

4. Open the appropriate run file.

5. If prompted, enter the storage location of batch(es) requested. Verify that the following information is accurate: sample number, standard name, class, batch number, matrix, protocol, method reference, LCS ID, initial volume, final volume and dilution factor. Make corrections as needed.

6. Enter the correct rinse time. Enter the appropriate analyst number.

7. Click on Check/Save. When the confirmation screen appears, verify that the file name and run number are the same. Choose to either Exit or Import another run and the run is imported and printed.

Routine Maintenance for the ICAP™ 6000 Duo Analyzer

A. Sample introduction system removal and cleaning: Remove and clean the sample introduction system when instrument performance declines (See **Figure 1**). Any adjustment to an instrument (replacement of parts, etc.) must be documented in the appropriate instrument logbook.

1. Remove spray chamber and nebulizer.

a. Unclamp spray chamber from spray chamber adapter by gently squeezing clamp with one hand while supporting the spray chamber with the other hand.

b. Gently remove the nebulizer from the spray chamber by pulling it out.

c. Disconnect the argon and sample tubing from the nebulizer by pinching the Luer lock and pulling the tubing off the nebulizer.

d. Clean the spray chamber if residue is observed coating the sides. If cleaning is necessary, remove the drain tubing from the spray chamber.

2. Remove and disassemble the torch

a. Gently pull the spray chamber adapter out of the torch assembly.

b. Unlock the torch assembly by turning it clockwise and remove it from the instrument.

c. Turn the injector tip assembly clockwise to separate it from the torch housing.

d. Gently pull the center tube out of housing to clean.

3. Prepare ultrasonic bath. Make sure the bath is at least ½ full with clean reagent water.

4. Clean torch, injector tip, and nebulizer.

a. Invert the torch in a 250-mL vacuum flask of 50% HCl and place in the sonicator for 10 minutes. After sonication, rinse the torch with reagent water. (Be careful not to get a lot of water down into the base of the torch.) Carefully dry the torch with a paper towel.

b. Place the injector tip in 50% HCl for 10 minutes. Rinse the injector tip with reagent water and carefully dry with a paper towel.

c. For a glass nebulizer, place the nebulizer in 50% HCl for 10 minutes. (Do not place nebulizer in the sonicator.) If there is a visible clog, carefully insert 0.13 diameter fishing line through the tip of the nebulizer to assist in removing the clog. Force the 50% HCl solution through the argon and sample inlets in the nebulizer and rinse with reagent water when finished. (Make sure all of the water is out of the argon cavity of nebulizer.) For a plastic nebulizer, use a syringe to force the 50% HCl solution through the nebulizer to clean and/or remove any clogs.

d. If the spray chamber needs to be cleaned, place it in the sonicator for approximately 5 minutes, and then rinse it out with matrix B rinse, followed by reagent water.

B. Reassemble the sample introduction system: (See **Figure 1**)

1. Reassemble the torch.

a. The o-rings on the metal torch mount must be inspected and replaced if any wear or damage is visible.

b. The quartz torch is pushed fully into the metal torch mount with a gently twisting pressure.

c. The circular “target” design on the torch **MUST** align with the circular notch on the torch mount.

d. Insert the injector tip fully into the center tube holder.

e. Insert the center tube assembly into the torch mount and rotate it counter-clockwise to lock it in position.

f. Mount the torch assembly back into the instrument by inserting it straight through the torch hole and coil, being careful not to disturb the quartz bonnet above the radial view lens.

g. Turn the assembly counter-clockwise to lock it into position.

h. Gently push the spray chamber adapter into the back of the center tube assembly.

2. Reassemble the spray chamber and nebulizer.

a. If removed for spray chamber cleaning, reattach the drain tube to the spray chamber.

b. With a twisting motion, insert the nebulizer into the spray chamber so that the collar is a tight fit.

c. Attach the sample and nebulizer gas tubing to the nebulizer.

d. Clamp the spray chamber to the spray chamber adapter.

3. Ignite the plasma.

C. Changing the pump tubing: Change pump tubing on the peristaltic pump when the tubing shows wear. Inspect all tubing to insure that it is secure and in good condition.

D. Documentation for instrument/analysis tag out and return to service.

NOTE: The following information is taken from 1-P-QM-QMA-9017325: In the event of an equipment failure, the following must be performed:

1. Document the nature of the failure in the maintenance logbook

2. Document how and when the defect was discovered

3. Notification of supervisor or responsible person who can decide on appropriate action to take

4. The instrument must be clearly tagged as *Out of Service*. The tag must contain the following information:

   a. Date taken out of service

   b. Employee who took the instrument out of service

   c. Reason for tagout

   Form 1-P-QM-FOR-9007909 is used for “tagging out”.

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken to bring the equipment back into service.

7. Results of the corrective action (i.e., system calibration within specifications, etc.)

8. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument. This is done on a case-by-case basis.

9. After repair, document whether the function has been fixed. Then determine if calibration or verification activities need to be performed before the instrumentation is put back into service.

Calculations

1. Final Result

   a. Water sample

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

All dilution factors must be recorded and used in the calculation. [To enter dilution data into the LIMS when multiple dilutions are used, a factor must be formed (Ex. 1), which contains no more than three figures for the volume or the aliquot (Ex. 2).]

Ex. 1. 50/.5 × 10/1 = 500/.5

Ex. 2. 50/.5 × 25/.5 = 1250/.25 = 125/.025

NOTE: The default units are µg/L

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{N}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{N}\right)}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\% \, Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)
Take either 4 identical aliquots (for 3 point MSA) or 2 identical aliquots (for one point MSA) of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution (for 3 point MSA) and spike the other aliquot at approximately the indigenous concentration of the sample (for one point MSA). Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots or

2 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^2 - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{\left[\sum (x_i - \bar{x})^2\right]\left[\sum (y_i - \bar{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ≥0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

Statistical Information/Method Performance

Generate MDLs and LOQs according to QA-SOP11892. Perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire digestion and analysis procedure. Compare and pool results to determine the final reporting MDL. The department supervisor maintains annual study data. The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

QC acceptance limits (MS, MSD, LCS and LCSD) are established as statistical limits (see QA-SOP11896 for guidance). Limits are evaluated every 6 months by the department and updated in LIMS by QA as directed by the department supervisor.

Quality Assurance/Quality Control

A. For 6010B, 6010C, and 6010D each digestion batch (up to 20 samples) must contain a method blank, LCS, and either an U, D, MS, MSD or an LCS/LCSD.

B. For 200.7, each digestion batch (up to 10 samples) must contain a method blank, LCS, and either an U, D, MS or an LCS/LCSD.

C. QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data.

D. Batch Quality Control - For the preparation and concentrations see T-MET-WI12063 and for the frequency, acceptance criteria and corrective action see tables I and II.

E. Raw data quality checks

1. Confirm that the batch and cover sheets are correctly labeled, dated, and signed where necessary. Review the batch sheet, project notes and lab notes with the incomplete list for special comments and due dates. Check that the run protocol has been selected correctly.

2. Check to see that the autosampler table printout is with the run and has a review signature from the analyst and run importer.

3. Refer to the calculation section of this SOP for calculations used for ICP analysis.

4. Refer to Tables I, and II for run and batch calibration and QC frequency, acceptance criteria and corrective action.

5. Each analytical run must have a QC review attached. All samples on the run must be listed on the QC review with notation as to whether the sample was verified or needed to be redigested/reanalyzed. The verifier must document on the QC review if any sample(s) were selected/deselected.

6. For spike levels of run QC see T-MET-WI12063

7. Spike levels of batch QC are available in the LIMS and on T-MET-WI9082.

8. LOQs are available in the LIMS and on T-MET-FRM8802.

9. Check to make sure that all results are below 90% of the linear range. If a sample reading is above 90% of the linear range, then reread the sample at an appropriate dilution. Verifiers footnote the coversheet indicating that all dilutions were performed correctly by comparing to the previous undiluted sample data.

10. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings <(−LOQ). Comments are added during verification to any non-detect sample readings that were diluted due to <(−LOQ).

11. For SPLP and TCLP samples, an MSA (method of standard additions) is required if:

    a. The sample concentration falls between 80% to 100% of the regulatory limits.

    b. If the SPLP or TCLP Matrix Spike (QA) recovers < 20%, all samples in the leachate batch must be reanalyzed using the method of standard additions for that analyte.

12. For all EW samples (samples from public drinking water sources); check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once. An automated email is sent to the Client Service Representative and the state for the analytes listed in QA-SOP11886 with the exception of lead and copper which follow the 90th percentile rule (the CSR tracks the lead and copper and notifies the supplier when necessary). Suppliers must be notified within 24 hours.

13. Check the internal standard (Yttrium) level for the entire run. If the Yttrium reading for any sample is < 50% or >130% of the reading for S0, then reread the sample at a dilution.

NOTE: The internal standard is added in equal concentration to all of the samples and standards via a dedicated line on the peristaltic pump. The analytical lines referenced to an internal standard report a corrected concentration value based on the ratio of analyte to internal standard intensities. All of the calculations for determining concentration are based off of Intensity Ratio (IR). The IR is defined as the background corrected intensity signal of the analyte line (Ia) divided by the internal standard value (Iis). IR = Ia/Iis

14. For EPA600 series samples, an ICV2 is analyzed immediately after the initial ICV. The average of the six total replicates is used with a requirement of ±5% accuracy and an RSD of <3%.

15. Check for high concentrations of interfering elements. Analytes must be flagged for possible interference if any interfering element concentration is greater than the level used for semi-annual IECs. Comments must be added during verification to any samples reading under the reporting limit that were diluted due to possible interference(s). For the iCAP 6000 series instrument (T70, #11016; T71, #16315; T72, #16417, T73, #18255) Si is monitored due to an interference on Pb. Pb data must be reread if Si is not within ±10% in CCVs.

NOTE: All samples requiring postspikes must be postspiked at 2 times the CRQL or approximately 2 times the indigenous level of the sample.

F. When raw data checks are complete, check the following:

1. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms. Any dilutions required have been calculated correctly and added to the reread/redigestion form. Specific instrument has been noted for client requirements if necessary.

2. Data for samples following Good Laboratory Practices (GLP) must be retained as permanent storage.

3. The data are uploaded to the LIMS via IDAT by the reviewer and are verified from the LIMS by the verifier.

G. Instrument detection limits are performed on a quarterly basis and method detection limits are performed on a yearly basis for each analytical instrument. Inter-Element Correction factors (IECs) are checked routinely every 6 months for each analytical instrument.

H. Verification process

1. Confirm that all required pieces of QC have been uploaded to the LIMS and are within specification. If there is partial QC on the current run and the samples have been analyzed more than once, check to see if there are associated runs in the hold bin waiting on additional QC to be verified.

2. In the LIMS, choose method of verification. (Metals verification by run, or verify by individual element.)

3. Ensure that all lab notes and project notes were followed.

4. Non-compliant data can be reported only after all required corrective actions have been taken.

5. When all of the elements are verified for a digest, verify the digest number. Associated tracking numbers and suite numbers are routinely auto-verified within hours of verifying all of the elements and digests on each sample.

Table I

Table I

QC requirements for SW-846 6010B, 6010C and 6010D (ICP Metals)

	Frequency	Acceptance	Corrective Action
Calibration	The calibration contains a blank and 1 standard.		
Initial Calibration Verification (ICV)	Must be analyzed immediately following Calibration Standards.	±10% of the true value. RSD must be <5% (6010B, 6010C, 6010D).	If the ICV is out of specification high for an analyte and the result is not < - LOQ, accept results that report as non-detect for affected analyte. Results for the affected analyte(s) > or = to the reporting limit must not be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be <3× IDL (6010B, 6010C) |ICB| must be <1/2 LOQ (6010D) If ICB is Out of Specification positive (+), accept results that are > 10X the ICB, or < reporting limit. If ICB is Out of Specification negative (-), only accept results that are > 10X ICB. (6010B, 6010C, 6010D).	Data for that analyte cannot be reported from the run for the affected samples (reanalyze the affected samples for that analyte) (6010B, 6010C, 6010D).

	Frequency	Acceptance	Corrective Action
Low Level Check (LLC)	6010B and 6010C: Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB. 6010D: Must be analyzed at the beginning of each run.	6010B: ±50% of True Value. Not applicable if sample concentrations are >10× the true value of the LLC. For LLC results >the high limit, samples <reporting limit can be accepted. 6010C:+/-30% of True Value. Not applicable if sample concentrations are < LOQ, or if sample concentrations are greater than the CCV (CCV must be within specification). 6010D:+/-20% of True Value. Not applicable if sample concentrations are < LOQ, or if sample concentrations are greater than the CCV (CCV must be within specification).	Data for that analyte cannot be reported from the run for the affected samples.
Interference Check Standard A and AB (ICSA/ICSAB)	6010B and 6010C: The ICSA must be analyzed at the beginning and end of each run immediately following the LLC. The ICSAB must be analyzed at the beginning and end of each run immediately following the ICSA. 6010D: ICSA only required (run on every run only at the beginning of each run immediately following the LLC).	±20% of the true value for analytes that are spiked. 6010B and 6010C: |ICS| or |ICSAB| must be <2× LOQ for analytes that are not spiked. 6010D: |ICS| must be < LOQ for analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze all samples requiring that element).

Table I (Continued)

	Frequency	Acceptance	Corrective Action

Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples.	±10% of the true value. RSD must be <5% (6010B, 6010C, 6010D)	If the CCV is out of specification high for an analyte and the result is not < -LOQ, accept results that report as non-detect for affected analyte. Results for the affected analyte(s) > or = to the reporting limit must not be reported from the run (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples	|CCB| must be <3× IDL (6010B, 6010C) |CCB| must be < ½ LOQ (6010D) If CCB is Out of Specification positive (+), accept results that are > 10X the CCB, or < reporting limit. If CCB is Out of Specification negative (-), only accept results that are > 10X CCB. (6010B, 6010C, 6010D).	Data bracketing the CCB for that analyte cannot be reported for the affected samples (reanalyze the affected samples in the bracketing blocks for that analyte) (6010B, 6010C, 6010D).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	|PB| must be <1/2 LOQ. For 6010B: Not applicable if analyte reading in the sample is > 20× the PB reading or <LOQ. For 6010C/6010D: Not applicable if analyte reading in the sample is > 10× the PB reading or <LOQ.	Redigest all associated samples.
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ.

Table I (Continued)

	Frequency	Acceptance	Corrective Action

Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken. RPD must be < 20%.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ. Redigest samples if RPD is out of specification
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±25% (6010B, 6010C, 6010D), as indicated by the client requirement. RPD must be <20%.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed. Flagged in the Data Package and in the QC summary.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	If the samples are >5× the LOQ the RPD must be <20%. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ. Not applicable if both samples are <LOQ.	Data is flagged in the QC Summary and/or in the data package.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike (s) are not within specification.	±25% of the true value.	The data is reported in the data package.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50× the MDL.	The percent difference must be <10%.	The data is flagged in the data package.

Table I (Continued)

	Frequency	Acceptance	Corrective Action

Samples		Results must be < 90% of the linear dynamic range, >-LOQ. RSD must be <20% for results >2x LOQ. Elements reported as non-detect are accepted if the ICV/CCV is out of specification high, and the sample is not < - LOQ.	Sample is diluted and reanalyzed. Sample is reanalyzed. Reanalyze for elements that do not meet this criteria.
Linear Range (LR)	Analyzed quarterly.	±10% of the true value	Samples reading greater than 90% of the calibration range must be reanalyzed.
Upper Linear Range	6010D only: Analyzed once per run.	±10% of the true value	Samples reading greater than 90% of the calibration range must be reanalyzed.
Mid Range Check (1/2 the Upper Linear Range)	6010D only: Analyzed once per run.	±10% of the true value	Reanalyze all elements > |CCV|.
Internal Standard	Added to samples in line by use of a mixing T.	Must be 50% -130% of the calibration blank.	Reanalyze at a dilution.

Table II

Table II
QC requirements EPA-600/R-94/111 (PW, EW, WW) ICP Metals

	Frequency	Acceptance	Corrective Action
Calibration	The calibration contains a blank and 1 standard.		
Initial Calibration Verification (ICV) ICV2	Must be analyzed immediately following calibration. ICV2 must be analyzed immediately after the ICV to attain the average of six replicates.	Avg of ICV and ICV2 must be ±5% of the true value. RSD for 6 replicates must be <3%.	If the ICV is out of specification high for an analyte and the result is not < - LOQ, accept results that report as non-detect for affected analyte. Results for the affected analyte(s) > or = to the reporting limit must not be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be < 3x IDL If ICB is Out of Specification positive (+), accept results that are > 10X the ICB, or < reporting limit. If ICB is Out of Specification negative (-), only accept results that are > 10X ICB.	Data for that analyte cannot be reported from the run (reanalyze all samples requiring that analyte).

	Frequency	Acceptance	Corrective Action
Low Level Check (LLC)	Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB.	Use statistical limits. Not applicable if sample concentrations are >10× the true value of the LLC. For LLC results >the high limit, samples <reporting limit can be accepted.	Data for that analyte cannot be reported from the sample.
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA must be analyzed at the beginning and end of each run immediately following the LLC. The ICSAB must be analyzed at the beginning and end of each run immediately following the ICSA.	± 20% of the true value for analytes that are spiked. |ICSA| or |ICSAB| must be <2× LOQ for analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze all samples requiring that element).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples.	±10% of the true value.	If the CCV is out of specification high for an analyte and the result is not < - LOQ, accept results that report as non-detect for affected analyte. Results for the affected analyte(s) > or = to the reporting limit must not be reported from the run (reanalyze).

Table II (Continued)

	Frequency	Acceptance	Corrective Action
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples.	|CCB| must be <3x IDL If CCB is Out of Specification positive (+), accept results that are > 10X the CCB, or < reporting limit. If CCB is Out of Specification negative (-), only accept results that are > 10X CCB.	Data bracketing the CCB for the affected analyte cannot be reported (reanalyze all samples in the bracketing blocks for that element).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	|PB| must be <1/2 LOQ or 2.2x MDL whichever is greater. Not applicable if analyte reading in the sample is >10× the PB reading or <LOQ. EW samples are not accepted for any reason if the PB is out of specification.	Redigest all associated samples. EW samples must be redigested.

	Frequency	Acceptance	Corrective Action
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less. *Note: The LCS is spiked at or below the MCL for all primary drinking water metals. See QA/QC section E.12. for MCL levels.	Use statistical limits or the method limit of ±15% (PW, EW)/ 20% (WW) ,as indicated by the client requirement. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken. EW samples are not accepted if the LCS is out of specification.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ. EW samples must be redigested.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 10 samples or less. *Note: The LCS is spiked at or below the MCL for all primary drinking water metals. See QA/QC section E.12. for MCL levels.	Use statistical limits or the method limit of ±15%(PW, EW)/ 20% (WW) ,as indicated by the client requirement. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken. EW samples are not accepted if the LCS is out of specification. RPD must be < 20%.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high, redigest samples that are greater than the LOQ. EW samples must be redigested. Redigest samples if RPD is out of specification

Table II (Continued)

	Frequency	Acceptance	Corrective Action
Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	Use statistical limits or the method limit of ±30%, as indicated by the client requirement.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	If the samples are >5× the LOQ the RPD must be <20%. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ. Not applicable if both samples are <LOQ.	Data is flagged in the QC Summary and/or in the data package.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	±15% of the true value.	Data is reported in the data package.

Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50× IDL.	The percent difference must be <10%.	Data is flagged in the data package.
Samples		Results must be < 90% of the linear dynamic range, >-LOQ. RSD must be <20% for results > 2xLOQ. Elements reported as non-detect are accepted if the ICV/CCV is out of specification high and the sample is not < - LOQ.	Sample is diluted and reanalyzed. Sample is reanalyzed. Reanalyze for elements that do not meet this criteria.
Linear Range (LR)	Analyzed quarterly.	±10% of the true value	Samples reading greater than 90% of the calibration range must be reanalyzed.
SIC (Spectral Interference Check)	200.7 Wisconsin Only: Analyzed once per day.	Interfering elements analyzed must be within 20%. Affected elements should read <LOQ.	Update interelement corrections so results are <LOQ and reanalyze SIC solution.
Internal Standard	Added to samples in line by use of a mixing T.	Must be 50% -130% of the calibration blank.	Reanalyze at a dilution.

Appendix I

Appendix I

Definitions and explanations of the codes and symbols used on the raw data

A. Sample table information

1. The run number.
2. The page number.
3. The tube number.
4. The sample number.

5. The first and second of four asterisks denote whether the sample is a background (U*), duplicate (D*), spike (R*), MSD (M*), post-digestion spike (UP), serial dilution (UL), or not a QC sample (**).

6. The weight to volume or volume to volume digestion ratio, consisting of the initial quantity of sample used and the final digest volume.

7. The dilution factor – Indicating if the digest solution was diluted prior to analysis. An undiluted sample is labeled DF1.

8. Digestion batch number – Assigned when designated samples are scheduled for preparation, this number is used to track samples and QC prepared together.

9. The protocol by which the data is reviewed (SW−846, EPA−600).

10. Date and time of the sample injection into the instrument.

11. The ICAP identification number.

Appendix I (Continued)

B. The ICP scans all of the method elements simultaneously during the analysis. The QC review lists all the samples on the run. The QC review lists elements verified, good phantom, and elements/phantom that are bad (need to be reread for run or batch QC). The reviewer or verifier documents on the QC review if any element(s)/sample(s) were selected/deselected.

C. The following are error codes in the iTEVA™ software.

1. S = Saturation – The concentration of the element is more than the detector can quantify.

2. K = The Elements Affected by a Saturated Element – The concentration listed is not accurate, and a more accurate result can be obtained by running the sample at a dilution.

D. Along with the average concentration (in ppm), the average intensity, %RSD and all three replicates are shown for each analyte. Internal standard values are intensities (cts/s).

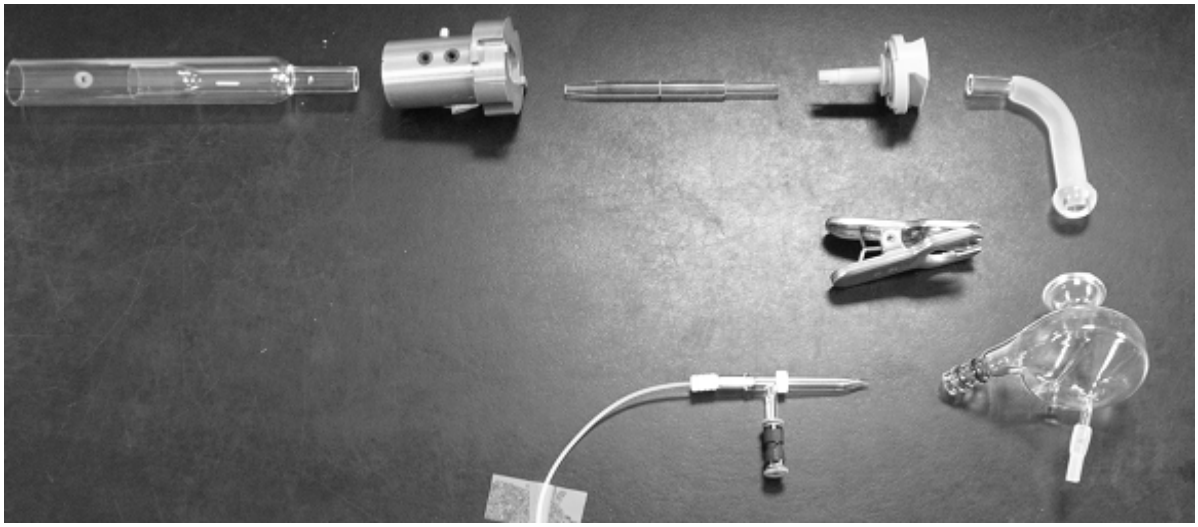
Attachment:
Figure 1

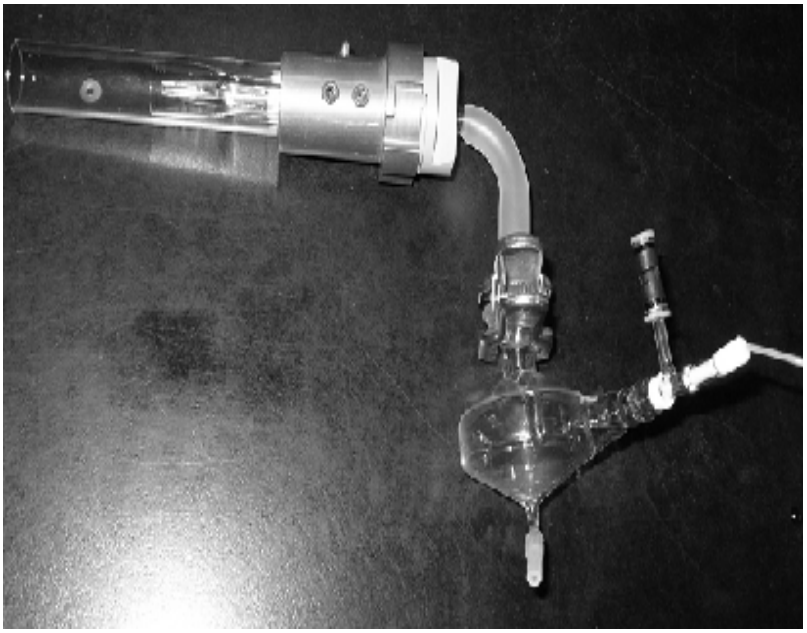
End of document

Version history

Version	Approval	Revision information
9	20.NOV.2015	
10	29.JUN.2017	

Figure 1





eurofins	Always check on-line for validity **Sample Preparation of Waters for Analysis of Total Recoverable Metals by Inductively Coupled Plasma Optical Emission Spectrometry**	Level: **Work Instruction**
Document number: **T-MET-WI8639**		
Old Reference: **1-P-QM-WI-9015133**		
Version: **16**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **03-DEC-2014**	Document users: **6_EUUSLA_Metals_ICP Prep, 6_EUUSLA_Metals_ICP Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis DOD - 1848, 10635

Revision Log

Revision: 16		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Historical/Local Document Number	No longer part of current procedure	Removed 5720
Reference	No longer part of current procedure	Removed CLP ILM02.1, ILM04.0, ILM05.2
Scope	No longer part of current procedure	Removed reference to CLP
Apparatus and Equipment	Reflect current procedure	Included DEENA to item 6.

Revision: 16	**Effective Date:** This version	
Procedure A	No longer part of current procedure	Removed Analysis 5720 (CLP) entire section
Procedure B	Clarification No longer part of current procedure	NOTE: Reworded text pertaining to the preparation of the PB and LCS for soluble metals analysis Removed Analysis 5720 (CLP)
Procedure C	Reflect current procedure	Included analysis 10635 (SW-846)
Procedure C.2	Reflect current procedure	Added DEENA temperature 90° to 95°C
Procedure C.5	Updated to current procedure	Replaced DI water with reagent water.
Quality Assurance/Quality Control	No longer part of current procedure	Removed reference to CLP digestion 5720 batch.

Revision: 15	**Effective Date:** Nov 27, 2013	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Sample Collection, Preservation, and Handling	Process change	Changed sample storage temperature from 4° ± 2°C to 0° to 6° C but not frozen, prior to digestion.
Procedure A	Reflect current procedure	Added text, to NOTE, pertaining to the blank and LCS must also be filtered if any samples are filtered due to insoluble matter present in the digested sample. Added NOTE, pertaining to using a smaller aliquot of the sample and bring sample to final volume, if contains high solids Added NOTE, pertaining to filtering unpreserved sample and preparing the blank and spiked LCS using filtered reagent water, for soluble metals analysis
Procedure A.1.a	Clarification	Added text pertaining to adding spike solution, after the sample has been poured.
Procedure A.2.a	Clarification	Added text pertaining to adding spike solution, after the sample has been poured.
Procedure A.2.a	Reflect current procedure	Added reference to Analysis 6966, 1643…… Deleted reference to SOP SOP-IO-014
Procedure A.2	Reflect current procedure	Added 2nd NOTE pertaining to Lab filtering.
Procedure B	Reflect current procedure	Added text, to NOTE, pertaining to the blank and LCS must also be filtered if any samples are filtered due to insoluble matter present in the digested sample. Added NOTE, pertaining to filtering unpreserved sample and preparing the blank and spiked LCS using filtered reagent water, for soluble metals analysis Added NOTE, pertaining to using a smaller aliquot of the sample and bring sample to final volume, if contains high solids.

Revision: 15		**Effective Date:** Nov 27, 2013
Procedure B.1.a	Clarification	Added text pertaining to adding spike solution, after the sample has been poured.
Procedure B.2	Reflect current procedure	Added NOTE pertaining to Lab filtering.
Procedure B.2.a	Clarification	Added text pertaining to adding spike solution, after the sample has been poured.
Procedure C	New equipment in use	Added entire section pertaining to DEENA Auto-digester.
Procedure C	Reflect current procedure	Added NOTE, pertaining to using a smaller aliquot of the sample and bring sample to final volume, if contains high solids. Added NOTE, pertaining to filtering sample, prep blank, and LCS if insoluble matter is present in the digested sample, or allow to settle by gravity. Added NOTE, pertaining to filtering unpreserved sample and preparing the blank and spiked LCS using filtered reagent water, for soluble metals analysis
Block Digester Instruction	Reflect current procedure	Clarified instruction steps. Deleted text pertaining to the difference between sample temperature and display temperature.
Quality Assurance/Quality Control	Reflect current procedure	Added reference to SOP 6966, 1643…for batch quality control requirements.

Reference

1. *Test Methods for Evaluating Solid Waste*, SW−846 Method 3005A, July 1992

2. *Chemical Hygiene Plan*, current version

Cross Reference

Document	Document Title
Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, …	Metals by Inductively Coupled Plasma Atomic Emissions Spectroscopy for SW-846 Methods 6010A/B/C (aqueous, solid, tissue), CLP 2.1(water/solid/tissue), CLP 4.0(water/solid/tissue), CLP 5.2 (water/solid/tissue) and EPA 200.7(aqueous)
1−P−QM−FOR−9009182	Working Instructions for Prep Solutions and Standards
1−P−QM−QMA−9015390	Demonstrations of Capability

Scope

This acid digestion procedure is used by the Metals Department of the Environmental Sciences Division at Lancaster Laboratories to prepare wastewater, surface water, and groundwater samples for measurement of total recoverable metals by inductively coupled plasma optical emission spectroscopy (ICP-OES) following SW-846 protocol.

This method is used whenever SW-846 Method 3010 is not requested or required for total metals.

Basic Principles

Samples are heated with nitric and hydrochloric acids with a substantial reduction in volume during digestion to dissolve metals.

Reference Modifications

1. A 50−mL sample aliquot and final volume is used instead of 100 mL to improve digestion throughput, conserve sample usage, and limit waste generation. Because all reagents are also adjusted so that concentrations are equivalent to a 100−mL aliquot, there is no impact on the data.

2. Ribbed watch glasses or reflux caps are not used during evaporation; samples are heated without watch glasses in non-metallic hoods to speed evaporation. No contamination trends have been observed in prep blanks evaporated without using watch glasses.

3. Samples are heated at 90º to 95°C on hotplates or Hotblocks, not 92° to 95°C as stated in ILMO4.0; hotplates cannot be maintained within 3°C range.

Definitions

1. ACS – American Chemical Society

2. D – Sample Duplicate

3. DOC – Demonstration of Capability

4. IDOC – Initial Demonstration of Capability

5. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate

6. LCSW– Laboratory Control Sample Water

7. LLENS - the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the LIMS.

8. LIMS – Laboratory Information Management Systems

9. LLI Sample ID – unique 7-digit number assigned to a client sample.

10. LOQ – Limit of Quantitation

11. MDL – Method Detection Limit

12. MS (R) – Matrix Spike

13. MSD (M) – Matrix spike duplicate

14. PB/PBW–Preparation Blank/ Preparation Blank Water

15. QC – Quality Control

16. Method Blank - equivalent to a Preparation Blank. A designated sample designed to monitor for sample contamination during the analysis process. A volume of reagent laboratory water is typically used to monitor water sample analysis, while solids blanks consist of a purified solid matrix or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

17. SOP- Standard Operating Procedure

18. U or US – unspiked background sample

Interferences

Not applicable to this procedure

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO_3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes or use the designated dispensing equipment.

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H_2SO_4 to avoid a violent reaction. Always use in a fume hood or use the designated dispensing equipment.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability for this or an equivalent procedure.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documentation of the critical steps in this procedure, over checking of data as well as an IDOC.

The IDOC and the DOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Refer to1-P-QM-QMA-9015390, for specific requirements. A DOC is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

Samples are collected in plastic containers and preserved to a pH of <2 with HNO_3. (Samples to be analyzed for soluble metals requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt and then preserved.) The pH is checked upon receipt and adjusted as necessary by Sample Support; samples that are pH adjusted at the lab must not be digested for a minimum of 24 hours. If samples fail to maintain a pH of < 2 the Client Service Representative is notified for further direction. Samples are stored at 0° to 6°C, but not frozen, prior to digestion. Samples must be digested within 6 months of collection. Digested samples are stored in plastic at room temperature and have a 6 month holding time.

Apparatus and Equipment

1. Polypropylene containers (digestion vessels) – certified clean and Class A equivalent

2. 250−mL beakers, 400−mL beakers (or other volumes as appropriate)

3. 100−mL graduated cylinders (or other volumes as appropriate)

4. 100−mL Class A volumetric flasks (or other volumes as appropriate)

5. 125−mL Nalgene bottles (or other volumes as appropriate)

6. Hot plates, Hotblocks, or DEENA, adjustable and capable of maintaining a temperature of 90° to 95° C

Reagents and Standards

For reagent preparation, shelf life, and storage conditions, see Form 1−P−QM−FOR−9009182.

1. Nitric acid, HNO_3 - Fisher, Trace Metal Grade, or equivalent. Store at room temperature and reevaluate annually.

2. Hydrochloric acid, HCl - Fisher, Trace Metals Grade, or equivalent. Store at room temperature and reevaluate annually.

3. Nitric acid (1:1) – Add 500 mL of HNO_3 to 500 mL of reagent water. Store at room temperature. Expires in 6 months.

4. Hydrochloric acid (1:1) – Add 500 mL of HCl to 500 mL of reagent water. Store at room temperature. Expires in 6 months.

NOTE: It is acceptable to prepare solutions using multiples of indicated volumes if exact ratios are maintained.

Calibration

Not applicable to this method.

Procedure

This SOP has been set up to outline the procedures for hotblock, hot plate, and auto-digester (see below). Choose the procedure that corresponds to the sample heating technique being used for sample digestion.

A. Hotblocks

NOTE: It is acceptable to reduce the volume of sample being analyzed as long as the sample reagent ratios are maintained.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

NOTE: When insoluble matter is present after digestion, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45-micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered reagent water.

See Hotblock Control Point Temperature Logbook to obtain control point temperature setting for the Hotblock being used for digestion. If necessary, adjust control point temperature to the proper setting as instructed below.

Analyses 1848 and 10635 (SW-846):

NOTE: The procedures for analysis 1848 and 10635 are equivalent as outlined below. Analysis 10635 is used only for SW-846 Update IV. When entering the batch number in the LIMS the “1” is omitted (i.e. use YYDDD0635###, where YY is the year, DDD is the julian day, and ### is the digest number).

1. Shake sample well. Transfer 50-mL of well−mixed sample into a 68−mL digestion vessel. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1−P−QM−FOR−9009182. For sample batch quality control requirements see SOP Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, …

2. Add 2 mL of (1:1) HNO3 and 5 mL of (1:1) HCl.

3. Heat the solution in a Hotblock at about 90° to 95°C until sample volume is reduced to between 15 and 20 mL, making certain the sample does not boil.

4. Allow to cool.

5. Adjust volume to the 50-mL mark on the digestion vessel with reagent water, cap and mix.

6. The sample is now ready for analysis.

B. Hot Plates:

NOTE: If boron (B) is requested on a sample, use Teflon vessels.

NOTE: It is acceptable to reduce the volume of sample being analyzed as long as the sample reagent ratios are maintained.

NOTE: When insoluble matter is present in the digested sample, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45-micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered reagent water.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

Analyses 1848 and 10635 (SW-846):

1. Shake sample well. Using a 50−mL graduated cylinder, transfer 50 mL of well−mixed sample into a 250−mL beaker. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1−P−QM−FOR−9009182. For sample batch quality control requirements see SOP Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, …

2. Add 2 mL of (1:1) HNO_3 and 5 mL of (1:1) HCl.

3. Heat the solution on a hot plate at 90º to 95°C until sample volume is reduced to between 15 and 20 mL, making certain the sample does not boil.

4. Allow to cool.

5. Transfer the solution to a 50−mL volumetric flask. Adjust volume to the 50-mL mark with reagent water and mix.

6. Transfer the solution to a 125-mL Nalgene container and cap.

7. The sample is now ready for analysis.

C. DEENA Auto-digester

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

NOTE: When insoluble matter is present after digestion, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45-micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered reagent water.

Analysis 1848 and 10635 (SW-846):

1. Shake sample well. Transfer 50-mL of well mixed sample into a 68-mL digestion vessel. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1−P−QM−FOR−9009182. For sample batch quality control requirements see. SOP Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, …

2. Verify the DEENA temperature is 90° to 95°C

3. Place samples into 20 position sample trays starting in position one. Place the trays into the DEENA.

4. Open the DEENA software. Click the Rack Definition button and input the total number of samples to be run.

5. Make sure all reagents have sufficient volume and that the transfer tubes are in the appropriate reagent. Make sure the waste beaker is clean and has 5-10 mL of reagent water in it.

6. Press the Green traingle (GO) button.

Block Digestor Instructions

1. Turn block digestor on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. PRESS and hold STAR (*) key.

4. The display shows the Set Point Temperature.

5. The digits can be changed to the desired value by pressing the up and down arrows keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 90° to 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations
Not applicable to this procedure.

Statistical Information/Method Performance
Not applicable to this procedure.

Quality Assurance/Quality Control
A method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample must be performed for every SW-846 digestion batch (analysis 1848 or 10635). A batch is 20 samples or less.

A method blank, sample duplicate, sample matrix spike, and laboratory control sample must be performed every CLP digestion batch (analysis 5720). A batch is 20 samples or less.

Each piece of batch QC is digested following the procedures in this SOP.

For sample batch quality control requirements see Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, …

End of document

Version history

Version	Approval	Revision information
16	03.DEC.2014	

eurofins	Always check on-line for validity **Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A/6020B(aqueous, solid, tissue) and EPA 200.8 (aqueous)**	Level: **Work Instruction**
Document number: **T-MET-WI11933**		
Old Reference: **1-P-QM-WI-9018443**		
Version: **7**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **29-JUN-2017**	Document users: **6_EUUSLA_Metals_ICP-MS Analysis, 6_EUUSLA_Metals_ICP-MS Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145

Table of Contents

Revision Log

Revision: 7	**Effective Date:**	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Enhancement	Added method 6020B
Throughout Document	Reflects updated company naming convention	Changed Parallax to LIMS
Throughout Document	New version of the method that is being supported	Added 6020B requirements (update V)
Definitions	Section not required	Removed section
Personnel Training and Qualifications	Enhancement	Added information on IDOC and DOC options.
Table 1	Clarification	Added to LCS/LCSD that the LCS is spiked at or below the MCL for all primary drinking water metals.
Table 1	Enhancement	Added new EW rule for the PB and LCS if they are out of specification, data cannot be accepted for any reason.

Revision: 6	**Effective Date:** Jan 15, 2015	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects re-identification of documents in EtQ No longer used New requirement	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers Removed CLP references. Added Uranium
Definitions	Clarification	Added that the CCV is prepared from the same source as the calibration standards.
Calculations	Clarification	Included information for 1-point MSA.

Reference

1. Method 200.8 (rev 5.4), Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry, USEPA 600/R-94/111 May 1994.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020, September 1994.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020A, February 2007.

4. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020B, Rev. 4, July 2014.

5. Agilent 7500 Series ICP-MS ChemStation (G1834B) Operator's Manual, Hardware Manual, Tuning & Application Handbook, and Maintenance Video (DVD).

6. ESI SC Manual.

7. Perkin Elmer Elan 9000 Hardware Guide, 2001& 2003.

8. Perkin-Elmer Elan Version 2.4 Software Guide, 2001 and Version 3.0 Software Guide, 2003.

9. Agilent 7700 Series ICP-MS MassHunter Workstation Guide and Hardware Maintenance Manual.

10. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
1-P-QM-FOR-9007858	Nonconformance Form
1-P-QM-FOR-9009076	Working Instructions for Preparation of ICP-MS Solutions and Standards
1-P-QM-PRO-9015511	Liquid Sample Preservation
1-P-QM-QMA-9017309	Determining Method Detection Limits and Limits of Quantitation
1-P-QM-QMA-9017313	Establishing Control Limits
1-P-QM-QMA-9017325	Instrument and Equipment Maintenance and Calibration

Purpose

The purpose of this SOP is to describe the proper analysis of aqueous and solid samples for metals by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) using Methods 6020/6020A6020B for aqueous, solid and tissue matrices or Method 200.8 for aqueous matrix. This SOP also outlines the proper operation and maintenance of the ICP-MS instrumentation and provides consistent guidelines for the evaluation of data

Scope

This procedure applies to analyses performed at Eurofins Lancaster Laboratories Environmental (ELLE) using ICP-MS for identification and quantitation of metallic constituents.

Limits of Quantitation (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

LOQs are subject to change without notification.

Routine Methods

Elements routinely analyzed on the Agilent 7500 and 7700 Series ICP-MS and/or the Perkin Elmer Elan 9000 ICP-MS include the following:

Analyte	Mass	Soil Analysis LIMS #	Water Analysis LIMS #
Li	7	10803	10804
Be	9	6127	6027
B	11	10801	10802
Na	23	6143	6043
Mg	24	6136	6036
Al	27	6123	6023
P	31	10805	10806
K	39	6140	6040
Ca	44	6129	6029
Ti	47	6147	6047
V	51	6148	6048
Cr	52	6131	6031
Mn	55	6137	6037
Fe	57	6134	6034
Co	59	6132	6032
Ni	60	6139	6039
Cu	63	6133	6033
Zn	66	6149	6049
As	75	6125	6025
Se*	78	6141	6041
Se**	82	6141	6041
Rb	85	13172	13171
Sr	88	6144	6044
Zr	90	10807	10808
Mo	98	6138	6038
Ag	107	6142	6042
Cd	111	6128	6028
Sn	120	6146	6046
Sb	121	6124	6024
Ba	137	6126	6026
Tl	203	6145	6045
Pb 206	206	6135	6035
Pb 207	207	6135	6035
Pb 208***	208	6135	6035

U	238	13502	13501

*Se 78 is the mass reported from collision cell ICPMS only

**Se 82 is the mass reported from non-collision cell ICPMS only

***For Pb masses 206, 207 and 208 are summed for calibration and analysis

Basic Principles

ICP-MS is an analytical instrument that uses the energy of an inductively coupled plasma to generate ions to be analyzed in the mass spectrometer. Several different means are utilized for sample introduction.

The Perkin-Elmer Elan 9000 uses a computer controlled peristaltic pump that delivers the sample from the autosampler into a cross flow nebulizer attached to a Scott spray chamber.

The Agilent 7500 and 7700 use a discrete sampling system that first loads the sample into a Teflon sample loop. The sample is then pushed by a carrier solution using a computer controlled peristaltic pump that delivers the sample from the loop into the nebulizer attached to an electronically cooled cyclonic spray chamber.

The Agilent 7500 and 7700 can analyze samples in 3 different modes of operation:

1. Mode 1 – no gas. This mode does not use a collision or reaction gas. Elements with a low atomic mass or that have no interferences can be analyzed using the “no gas” mode.

2. Mode 2 – Helium (He) mode. This mode uses He as a collision gas for reliable, predictable removal of unknown matrix interferences. No new interferences are formed in the cell, and no analytes are lost by a reaction.

3. Mode 3 – Hydrogen (H2) mode. This mode uses H2 as a reaction gas, since it reacts quickly and efficiently with the Argon-based interfering species, but reacts slowly or not at all with the analyte of interest. In this mode, interferences can be reduced to the level of baseline noise, allowing lower detection limits to be achieved for some difficult elements.

Interferences

ICP-MS interferences include isobaric elemental interferences and polyatomic ion interferences derived from the plasma gas, reagents, and/or sample matrix. The Agilent 7500 and 7700 utilize helium as an inert collision gas to reduce or eliminate many types of polyatomic ion interferences.

Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for using internal standardization.

Memory interference is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time of the sample introduction system overcomes any memory interference.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood.

Hydrofluric acid (HF) – This acid is very toxic and absorbs through the skin without pain. Wear double nitrile gloves and handle the solution in a fume hood with assistance from your supervisor. If you spill any HF on your skin IMMEDIATELY FLUSH the affected area with water and dial 1-1-1 immediately. The initial rinse must last for 5 minutes, followed by a liberal application of calcium gluconate gel.

Double nitrile gloves must be worn while the gel is applied and massaged into the affected area. EMERGENCY CARE TREATMENT IS REQUIRED FOR ALL HF EXPOSURES. SEEK MEDIAL ATTENTION IMMEDIATELY (DIAL 9-1-1 OR GO TO THE NEAREST EMERGENCY ROOM.)

For small HF spills (a few drops) on the counter top or floor, dilute with water to reduce fumes given off, dust the spill with boric acid powder, and then follow up with copious water rinse. Boric acid

powder is stored on the prep room shelf. **For all other HF spills, immediately evacuate the area and dial 1-1-1; full protective equipment is required to treat large HF spills.**

Hydrogen Peroxide 30% (H2O2) - This oxidizer can cause skin burns. Always use in a fume hood.

Nitric acid (HNO3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A , soda ash or equivalent, to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf. Soda ash is located in the stairwell adjacent to the prep room.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the sequence editor to set up the run, perform calculations, interpret raw data, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an IDOC (Initial Demonstration of Capability).

The IDOC consists of four laboratory control samples (LCS) that are carried through all steps of the prep and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Sample Collection, Preservation, and Handling

A. Aqueous samples – Aqueous samples are collected in plastic or glass containers with the exception of Drinking Water samples which are collected in 1-L plastic or glass containers. Aqueous samples are preserved with nitric acid and stored at 0° to 6°C not frozen. Samples must be digested within 180 days of collection for all Methods.

B. Solid samples – Solid samples are collected in glass containers and stored at 0° to 6°C not frozen. Samples must be digested within 180 days of collection.

C. pH Adjustment

1. Upon receipt at the laboratory, Sample Storage personnel check the pH of water samples. If the pH is greater than 2, the pH of the sample is adjusted to a pH less than 2 following the protocol outlined in 1-P-QM-PRO-9015511.

2. Samples requiring pH check immediately before digestion and analysis must be tested and if pH is greater than 2, the client service representative is notified. The client service representative must notify the client for direction on how to proceed with the sample (i.e. proceed as is or add more acid.)

3. Dissolved Metals: Samples to be analyzed for metals requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt and then for aqueous samples**,** samples are collected in plastic containers and preserved to a pH of <2 with HNO_3.

4. Solid samples require no chemical preservation.

D. Storage - Store sample digestates in plastic bottles at room temperature. Store standards and digestates separately.

E. Sample Discard - The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and verified in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

A. The following is a list of the hardware used in the Agilent 7500, Agilent 7700 and Perkin-Elmer Elan 9000 ICP−MS systems.

1. Spectrometer

a. The Agilent 7500ce is an inductively coupled plasma mass spectrometer. The sample introduction system consists of an ESI FAST discrete sampling system, Teflon nebulizer and electronically cooled spray chamber attached to a concentric quartz tube plasma torch.

b. The Perkin-Elmer Elan 9000 is an inductively coupled plasma mass spectrometer. The sample introduction system consists of a Ryton crossflow nebulizer on a Scott spraychamber attached to a concentric quartz tube plasma torch.

c. The Agilent 7700x is an inductively coupled plasma mass spectrometer. The sample introduction system consists of an ESI FAST discrete sampling system, glass concentric or Teflon nebulizer and electronically cooled spray chamber attached to a concentric quartz tube plasma torch.

2. Autosampler

a. The Agilent 7500 and 7700 systems use an ESI SC-8 FAST autosampler. The autosampler parameters for each automated run are entered into the Edit Sample Log Table interface editor in the ChemStation software (7500) or in the Sample List in MassHunter software (7700).

b. The Perkin-Elmer AS-93*plus* autosampler has capacity for 149 samples and 8 standards. The autosampler parameters for each automated run are entered into the sample window of the Elan software.

3. Coolflow

a. The Agilent 7500's G3292A Recirculating Chiller is set up to deliver cooling liquid to the ICP−MS at a regulated pressure of 60 psi.

b. The Perkin-Elmer's Polyscience cooling system is set up to deliver cooling liquid to the ICP−MS at a regulated pressure of 50 psi.

c. The Agilent 7700's G1879B Heat Exchanger is set up to deliver cooling liquid to the ICP–MS at a regulated pressure of 30 psi.

4. Computer

a. The Agilent 7500ce is controlled by a Windows–based IBM compatible PC with ICP-MS ChemStation software installed.

b. The Perkin-Elmer Elan 9000 is controlled by a Windows–based IBM compatible PC with Elan Version 2.4 or Version 3.0 ICP–MS software installed.

c. The Agilent 7700x is controlled by a Windows–based IBM compatible PC with MassHunter software installed.

5. Vacuum Pumps

a. The Agilent 7500ce and 7700x both have a 3-stage vacuum system. A rotary pump evacuates the interface chamber; a turbomolecular pump evacuates the ion lens chamber; and a turbomolecular pump evacuates the analyzer chamber.

b. The Perkin-Elmer has a rotary vane vacuum pump hooked up to the region between the sampler and skimmer cones. This pump maintains the interface region at approximately 4 torr of vacuum. In the quadrapole region, there is a dual inlet turbo molecular pump. This pump maintains the ion optic region at a vacuum of approximately 8×10^{-4} torr, and the mass filter region at a vacuum of approximately 1×10^{-5} torr.

6. Ultrasonic bath

7. Polishing paper (#400 and #1200)

8. Alumina powder

B. The following is a list of the apparatus necessary for the setup of an ICP–MS run for analysis:

1. ICP-MS run cover sheets. Cover sheets are generated using the IDAT Sequence Editor program in LIMS.

2. Test tube racks

3. 17 × 100−mm polystyrene tubes

4. Graduated 15−mL polypropylene tubes and caps

5. 50-mL polypropylene screw cap tubes

6. FilterMate 2 µm filters and plungers

7. 1 × 100 10−mL sterile disposable syringes

8. 25−mm syringe filters, PTFE, 0.45 µm

9. 30−mL polypropylene medicine cups

10. Adjustable electronic hand−held pipettes (10 - 5000 µL) - Fisherbrand® or equivalent.

NOTE: For routine operation, calibration, and maintenance of adjustable electronic hand-held pipettes, see 1−P−QM−QMA−9017325.

11. Fixed volume hand-held pipettes (25 – 2000 µL) - Eppendorf or equivalent.

NOTE: For routine operation, calibration, and maintenance of fixed volume hand-held pipettes, see1−P−QM−QMA−9017325

Reagents and Standards

Refer to Form 1−P−QM−FOR−9009076 for reagent and standard information and the preparation of the following standard and solutions.

A. ICP MS Standards

B. ICP MS Initial and Continuing Calibration

C. ICP MS LOQ Check Standard Solution (LLC)

D. Interference Check Solutions

E. Rinse/Carrier, Tuning and Calibration Solutions

F. Internal Standard Solution

G. PDS Solutions

H. Matrix Matched Standards

I. General Acids and Chemicals

Calibration

A. Initial Calibration.

1. For the preparation and concentrations of calibration blanks and calibration standards see 1-P-QM-FOR-9009076.

2. For the frequency, acceptance criteria and corrective action see tables I, and II.

B. Initial Calibration Verification (ICV).

1. For the preparation and concentrations of ICV standard see Form 1-P-QM-FOR-9009076.

2. For the frequency, acceptance criteria and corrective action see tables I, and II.

C. Continuing Calibration Verification (CCV).

1. For the preparation and concentrations of CCV standard see Form 1-P-QM-FOR-9009076.

2. For the frequency, acceptance criteria and corrective action see tables I, and II.

D. Low Level Check Standards (LLC)

1. For the preparation and concentrations of LLC standards see Form 1-P-QM-FOR-9009076

2. For the frequency, acceptance criteria and corrective action see tables I, and II.

E. ICSA/ICSAB

1. For the preparation and concentrations of ICSA/ICSAB standards see Form 1−P−QM−FOR−9009076

2. For the frequency, acceptance criteria and corrective action see tables I, and II.

3. As of 10/10/2012 due to the high total dissolved solids in these interference check solutions a rinse sample is analyzed after each ICSA/ICSAB pair for 6020A update 4 and 6020B update 5.

Procedure

A. Setting up an ICP−MS run

The procedure is the same for both the Perkin Elmer Elan 9000, Agilent 7500ce and Agilent 7700x instruments.

1. Determine the batches to be analyzed and determine any special requirements by viewing lab notes and/or project notes that are with the batch paperwork .

NOTE: An ICP-MS run typically contains no more than 60 tubes; however, it is acceptable to use more tubes than this, especially for clean matrix samples (i.e., undigested drinking water samples).

2. Open the Sequence Editor program from the IDAT menu in eLIMS.

3. Select “ICP-MS” in the Instrument Method drop down list.

4. Select the appropriate digest type and load the list of batches.

5. Select the appropriate template and batch and add the batch to the template.

6. The following information is added automatically by the Sequence Editor program.

   a. Sample names include:

      (1) PBW – Prep blank (water)

(2) LCSW – Laboratory control sample (water)

(3) LCSDW – Laboratory control sample duplicate (water)

(4) PBS – Prep blank (solid)

(5) LCSS – Laboratory control sample (solid)

(6) LCSDS – Laboratory control sample duplicate (solid)

(7) Lancaster Laboratories' sample number

(8) CCV – Continuing calibration verification

(9) CCB – Continuing calibration blank

(10) LLC – Low level check

(11) ICSA – Interference check standard – A

(12) ICSAB – Interference check standard - AB

(13) S0 – Calibration blank

(14) S1 – Calibration standard 1

(15) CCS – Carryover Control Standard

NOTE: A single point calibration, consisting of a blank (S0) and one standard (S1), is used for all analysis except samples requiring a multipoint calibration. When a single point calibration is used, the upper standard is at 100 ppb for all analytes reported by the method. For multipoint samples, a multi-point calibration is used consisting of a blank, 25 ppb, 50 ppb and 100 ppb standards.

b. Initial volume (IV) – The sample aliquot digested. IV is not included on the cover sheet but is pulled from LIMS during import.

c. Final volume (FV) – The final sample volume after digestion. FV is not included on the cover sheet but is pulled from LIMS during import.

d. Dilution factor (DF) – The dilution factor of the sample prepared at the time of analysis, needed to bring the sample into the linear range of the instrument, to negate a matrix effect, or for serial dilutions. If dilutions other than the default dilution are required the analyst must change the DF for affected tubes prior to saving the sequence.

e. Batch No. – The batch number of the sample. The Sequence Editor Program automatically inserts the batch number.

f. Protocol – The protocol used to review the data for specific method requirements in the IDAT database. Each template in Sequence Editor has a default protocol. If samples require a different protocol it must be changed for the appropriate tubes prior to saving the sequence.

g. SDG – Sample delivery group number for data package samples. SDG is not included on the run cover sheets.

h. Comments – Any description of the sample (from prep logs), status of the sample (i.e., RUSH, Promised) and due date are recorded here. The list of elements needed for the run is automatically inserted on the cover sheet by the Sequence Editor Program.

i. Lot numbers – Lot numbers must be recorded by the analyst at the start of the analytical run.

7. When setting up a run using the Sequence Editor Program, Batch QC (i.e., PB, LCS, background, duplicate, matrix spike, matrix spike duplicate, post-digest spike, and serial dilution) are automatically placed in a block of ten or fewer samples. If there is an LCS and LCSD, they are automatically placed one after the other.

a. ICV/ICB must be analyzed immediately after the calibration curve.

b. CCV/CCB must be run after every ten analytical samples.

c. LLC, ICSA, ICSAB, CCV, CCB immediately follow the ICV/ICB and must conclude each run.

NOTE: As of 10/11/2012 for all DOD protocols, it is not necessary to analyze the ending ICSA/ICSAB and LLC check samples. This is for DOD only!

d. Any deviations from protocol must be noted in the Comments Section of the cover page.

e. Verify that all information has been entered correctly.

f. Save the file, this automatically generates the cover sheet.

g. Add batch location and any comments to the cover sheet.

h. Print the cover sheet.

B. Pouring an ICP-MS run

The procedure is the same for the Perkin Elmer Elan 9000, Agilent 7500ce and Agilent 7700x instruments.

It is important to minimize any chance of contamination, to both yourself and the samples. Keep your hands and the work area clean at all times. Wear appropriate PPE at all times.

NOTE: See Form 1-P-QM-FOR-9009076for standards and solutions used during the analytical run.

1. Choose the appropriate run sheet. Record the following on the first page of the ICP-MS run sheet: initials, employee number, and the date.

NOTE: When retrieving the batch from the shelf for the first time, record the batch location on the cap of the prep blank in black sharpie. This saves time when returning the batch to the proper location after analysis. To prevent more than one batch being assigned to the same location a card system is used – when no batch is assigned to a location the card is placed on the shelf with “OPEN” facing up; when a batch is assigned a location on the shelf the card is placed on the shelf with “IN USE” facing up. Leave the card on the shelf whenever the batch is removed for pouring or analysis. When samples are discarded the corresponding card is flipped to “OPEN”.

2. Batches prepared in hotblocks (50-mL digestion cups only)

a. The only tubes that need to be poured for the initial run are the PDS (UP) and serial dilution (UL).

b. All other samples remain in the 50-mL digestion cups for analysis.

3. Samples that need to be poured into test tubes.

    a. Obtain the appropriate number of tubes.

    b. Number each tube with the sample number, dilution factor and batch location.

    c. Place them in test tube racks.

    d. The analyst must ensure that tubes are labeled correctly, that dilutions are performed accurately, and that the dilutions are recorded on the run cover sheet.

4. Post−digest spike (PDS)

    a. PDS is required for each batch (sample volume permitting).

    b. A PDS is prepared by placing 0.2 mL of the appropriate PDS solution into a 14 mL graduated test tube and bringing to a volume of 10 mL with the background sample.

    c. Record the volume and lot number of PDS solution used as well as the final volume in the comments column of the ICP−MS run sheet.

5. Serial dilution (SD)

    a. Prepare the SD by diluting the background sample at a dilution that is equal to 5× the dilution factor of the background sample (i.e. if Bkg = DF1, SD must = DF5; if Bkg = DF5, SD must = DF25).

    b. Document the volume of background sample used as well as the final volume in the comment section.

6. Filtering samples

    a. Samples that are cloudy or have particulate suspended in solution must be filtered prior to analysis to prevent clogging of the sample introduction system, which causes run failures and instrument down time to correct the problem.

    b. Samples that are digested using the 50 mL hotblock tubes need to be filtered using the FilterMate filtration devices and plunger.

c. Attach a filtration device to the plunger and very carefully insert the filter into the hotblock tube until the filter reaches the bottom of the digestion tube.

d. Remove and discard the plunger.

e. An alternative for samples in any type of vessel is to filter using a 10−mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter.

f. If any samples are filtered, the prep blank must also be filtered.

g. Document all filtrations on the run cover sheet.

NOTE: It is not necessary to filter all samples (in hotblock tubes only) that contain particulate, as long as all of the particulate is settled to the bottom of the tube below the 5 mL mark in 50 mL digestion vessels. The sampling depth of the autosampler probe is set such that the tip of the probe is approximately 1 cm above the bottom of the digestion tube.

7. Verify that all samples in the hotblock tray are in the correct position and/or pour each sample (or sample filtrate) into the appropriate tube. Usually, the order of the batch QC is PB, LCS, (LCSD), Bkg, PDS, DUP, MS, (MSD), and SD. Most importantly, the actual positions of the samples in the tray must match the autosampler table used for analysis.

NOTE: Immediately prior to beginning analysis, the analyst must visually verify that each sample is in the position indicated in the autosampler table used for the run.

8. If not being analyzed immediately, cover any poured tubes with plastic wrap to prevent contamination of the samples (hotblock tubes and dilutions prepared in graduated test tubes must be capped tightly except during pouring and/or analysis).

9. Return samples to sample storage, being sure to place the batch back into the location recorded on the batch sheet.

10. NOTES:

a. A PDS and a SD are performed on one sample in each digestion batch. Typically, the background sample is chosen. If the batch QC is split between two samples, the PDS is performed on the background sample accompanied by a matrix spike; the SD is performed on the background sample accompanied by a matrix duplicate. If sample volume is limited, it is acceptable to use the duplicate for the PDS and SD.

b. Batches with only field blanks or equipment blanks do not need a PDS or a SD.

c. Air filter batches need only a SD on one sample in the batch (a PDS is not required).

d. "As Received" samples are analyzed with a blank and LCS, LCSD (prepared by the analyst). These "batches" are assigned a batch number by the analyst and recorded into the departmental LLENS system and is uploaded to LIMS.

e. Documentation is of utmost importance. Verify all entries.

Instrument Operations

A. Startup, Shutdown and Status of the Agilent 7500ce ICP-MS

NOTE: When referring to menus in the procedure below "ChemStation menu" refers to the pull down menus at the top of the ChemStation software window. Other menus are referred to by the name that appears in the title bar of any window that opens within the ChemStation software.

NOTE: ChemStation limits file and folder names to a maximum of 8 characters. Any time the user is prompted by this procedure to save a file or folder while using ChemStation the file or folder name must not exceed 8 characters. Refer also to pages 4-78 in the ChemStation Operator's Manual for a list of special characters that must not be used when naming files and folders within ChemStation.

1. To open the ChemStation software:

   a. If not already active, double−click on the "ICP-MS Top" icon on the computer desktop to start the software. Alternatively, single click on the "ICP-MS Top" icon in the Quick Launch bar.

   b. Activate the Instrument Control window by selecting the appropriate icon at the top of the screen or by selecting *Instrument >> Instrument Control* from the ChemStation menu.

2. Startup of the Agilent 7500ce ICP−MS:

   a. There are three states from which the Agilent 7500ce is started: unplugged (i.e. cold), shutdown mode, and standby mode. The current mode is displayed in the title bar

of the Instrument Control window. See Chapter 3 of the ChemStation Operator's Manual for more detailed instructions.

b. Check that the waste drain vessel is not full, and has adequate space remaining to complete the automated run without overfilling.

c. Check that the sample, internal standard, and drain lines are attached to the peristaltic pump and that all tubing is connected properly.

d. Check to see that there is adequate internal standard solution to complete the entire automated run.

e. Check to see that the autosampler rinse vessel has sufficient rinse for duration of the automated run. If not, refill with the appropriate rinse solution. The carrier solution probe and the internal standard probe are placed in a bottle containing reagent water.

f. If a cold startup is required refer to p. 3-3 in the ChemStation Operator's Manual. Continue to the next step only after confirming that the instrument is in Shutdown mode.

g. To start the instrument from Shutdown mode select *Vacuum* >> *Vacuum* On in the Instrument Control window. Select "Yes" to confirm this action. Once the vacuum chamber reaches the correct pressure of approximately 5 x 10^{-4} Pa the instrument goes into Standby Mode.

h. To start the instrument from Standby Mode, first verify that the instrument coolflow is on. *Select Plasma* >> *Plasma On* in the Instrument Control window. Once the interface vacuum has reached approximately 4.5 x 10^{-2} Pa and the plasma is lit, the instrument switches to Analysis mode.

i. Allow the instrument to warm up for at least 15 minutes if the plasma has been off for 15 minutes or less. Allow at least 30 minutes of warm up time if the plasma has been off for more than 15 minutes.

B. Agilent Tuning and set up of the ChemStation software

1. Open the ChemStation software if not already open.

2. Analyzing the Daily EPA Tune sample

a. Tuning must only be done after the instrument has been allowed to warm up as described above in Instrument Operations, section A.2.i. In the ESI software "FAST Control Enabled" must be unchecked during analysis of the EPA Tune sample.

b. Place the carrier probe into the EPA Tuning Solution (10 ppb Ba, Cd, Ce, Cu, In, Mg, Pb, Rh and U). The internal standard line must remain in the reagent water bottle, and the analyst must ensure that there are no air bubbles in either line during analysis of the tuning solution.

c. Select *Sequence>> Load and Run Sequence* from the ChemStation menu. Choose "EPA_Tune.S" from the file list and click "OK".

d. In the Start Sequence window, type in your user ID (i.e., abc01234) as the operator name. Note that the default Data Batch Directory path is "C:\ICPCHEM\1 \DATA\[NAME].B\", where [NAME] is the folder name assigned by the Agilent software. Change the directory path to "D:\ICPCHEM\1\DATA\YYDDD##.B\", where "YY" is the two digit year, "DDD" is the Julian day, and "##" is the tune number. Once these changes have been made, click "Run Sequence".

e. Once the analysis of the Tuning sample is complete, a Tune Report prints. Verify that the Tune Result is "Pass" and use the Offline Data Analysis to generate a data file for import into LIMS. This file must then be imported using the IDAT ICPMS Tune Data program in LIMS prior to importing sample runs for that day. Keep the passing Tune Report for reference and recycle any previous Tune report(s).

NOTE: If the Tune Result is "Fail," repeat steps c. and d. above. If a second Tune failure is encountered, evaluate whether maintenance or optimization are required.

f. Once the Tune data file is imported the Tune Report is automatically appended to the run report for any additional runs.

NOTE: The EPA Tune sample is analyzed once at the beginning of each day **OR** after any maintenance, before starting any analytical runs (even if the EPA Tune sample has been analyzed earlier on the same day).

g. Tuning and optimization of the Agilent 7500ce ICP-MS

NOTE: The manuals and software refer to the various instrument optimization parameters as "Tuning". This differs from the EPA Tuning described in this section.

(1) Tuning and optimization are performed on an as needed basis, either as corrective actions for poor instrument performance or as required steps following instrument maintenance.

(2) Refer to the Agilent 7500 Tuning Manual for complete details on instrument tuning procedures.

3. After analysis of the EPA Tune sample is complete, place the internal standard line into the internal standard solution. The carrier probe must now be placed in the Carrier Solution bottle. Before commencing sample analysis “FAST Control Enabled” must be checked in the ESI software.

4. Load the method to be used for analysis by selecting *Methods* >> *Load* from the ChemStation menu. Double-click the method that you wish to use in the Select Method File window that appears. In the Select Calibration File window, verify that the selected calibration file has the same name as the method you selected; then click “OK”.

5. Next load, edit, save and print the sample sequence. Procedure 5.a. describes how to do this using the online Edit Sample Log Table interface in the ChemStation software. Procedure 5.b. describes how to enter sequences using the Offline version of the Edit Sample Log Table and how to utilize the Chained Sequence.

a. How to enter a sequence using the online Edit Sample Log Table interface:

(1) Open a sequence for editing by first selecting *Sequence* >> *Load* from the ChemStation menu.

(2) Choose an appropriate file from the list; then click “OK”.

(3) If it is a new sequence you must rename the file.

(4) Select *Sequence*>> *Save* from the ChemStation menu and enter an 8 digit file name using the Julian day, digest, and last digit from the batch number.

(5) Next select *Sequence* >> *Edit Sample Log Table* from the ChemStation menu.

(6) Double–click on the “1” in the first row in the column on the far left of the Edit Sample Log Table window.

(7) Right click anywhere in the sequence and select “Load list from CSV file”.

(8) Navigate to the IDAT Sequences folder and select the appropriate batch number and click Open.

(9) If you need to change the method choose a method by double-clicking on the first row in the “Method” column.

(10) Choose a method from the list; then click “OK”.

(11) Select all the cells in the “Method” column starting with the cell in the first row down to the cell in the last row of the sequence.

(12) Right-click on the selection; then select “Fill down” to copy the method from the first cell into the rest of the selection.

(13) Note that the correct sample positions for the SC-8 autosampler are not currently populated by the Sequence Editor program.

(14) Select an appropriate template from the “Default Positions for SC-8” folder in the taskbar and copy and paste the positions into the sequence.

(15) Select the S0 through the last sample in your run, press Ctrl-C, and then switch to the Edit Sample Log Table window, select the cell that corresponds to the calib blank in the Vial column, and press Ctrl−V.

(16) Add any additional rinse or conditioning tubes to the beginning of the sequence as needed.

(17) Once you have verified that all information has been entered correctly, click “Print” in the lower right corner of the Edit Sample Log Table interface.

(18) Click “OK” to return to the main ChemStation window.

(19) Then select *Sequence* >> *Save* from the ChemStation menu. When prompted as to whether you wish to overwrite the existing file select “Yes” (the file name already corresponds to the batch number on the cover sheet).

(20) After double checking the entries on the printed Sample Log Table, initial the top of the printout.

b. How to enter a sequence using the offline Edit Sample Log Table interface and using the Chained Sequence in ChemStation:

(1) The Chained Sequence function in ChemStation allows the analyst to add a sequence to a queue while a run is in progress. This allows the next run to commence as soon as the current run completes minimizing gap times between runs. It is also used to allow for unattended analysis during an off shift taking full advantage of the multibatch capacity of the SC-8 autosampler.

(2) Setup your first run using the instructions above in part a, steps (1) through (20).

(3) Commence analysis by selecting “Chained Sequence” then “Edit and Run” from the ChemStation menu.

(4) Add the appropriate sequence, and edit the Data Batch and Tune file fields.

(5) Once you have loaded the autosampler, click “Run” on the Edit Chained Sequence window to begin analysis. The autosampler immediately moves to the first vial in your sequence.

(6) While analysis of the first run continues, setup additional sequences using the offline version of the Edit Sample Log Table and add them to the Chained Sequence by editing the Sequence File, Data Batch and Tune file fields as needed.

(7) To open the offline Edit Sample Log Table chose “ICPMS Offline ChemStation” and “Edit Sample Log Table” from the Programs folder in the Start menu.

(8) Click the “Load” button at the bottom of the screen.

(9) Follow the “Shortcut to Sequence on D” to get to the folder where ChemStation sequences are stored and open an appropriate sequence. If the batch is being analyzed for the first time the file must be immediately renamed; click the “Save” button at the bottom of the Edit Sample Table window and enter an 8 digit file name that corresponds to the batch number on the cover sheet. Do

not overwrite any sequence that has already been added to the chained sequence.

(10) Double−click on the “1” in the first row in the column on the far left of the Edit Sample Log Table window. Continue to edit and print the file as outlined in steps (7) through (14) in procedure 5a above.

(11) Save the sequence by clicking the “Save” button at the bottom of the window. When prompted as to whether you wish to overwrite the existing file select “Yes” (the file name already corresponds to the batch number on the cover sheet).

(12) Click “Exit” at the bottom of the window to exit the offline Edit Sample Log Table.

(13) Add the sequence to the Chained Sequence and load the autosampler for the newly added sequence. Be sure to use the next blank line in the Chained Sequence window each time you add a new sequence.

(14) After double−checking the entries on the printed Sample Log Table, initial the top of the printout.

6. Before beginning analysis, the analyst must visually verify that all sample numbers and vial positions on the printed Sample Log Table correspond to the samples and standards as loaded on the autosampler prior to initialing the top of the printout. If changes are needed during the run sequence the analyst must print the modified Sample Log Table, and confirm that the changes on the printout match what is on the autosampler. Keep the original and any modified Sample Log Table printouts with the run until verification is complete.

7. Verify that the appropriate Sequence is loaded by viewing the selections shown in the drop down menus across the top of the ChemStation software. Access the drop−down menus to quickly select the appropriate files if they are incorrect. Alternatively verify the correct sequence(s) is loaded in the Chained Sequence window.

8. P lace the standards and samples in the appropriate locations on the FAST SC-8 autosampler.

a. The autosampler positions (“Vial” column in the Sequence) are defined as follows:

Standard ID	Vial	Sequence #	Tube type

S0 (Blank)	2101	1	17 × 100−mm Tubes
S1 (Standard 1)	2102	2	17 × 100−mm Tubes
ICV	1101	3	50-mL Screw Cap Tubes
ICB/CCB	1102	4/9	50-mL Screw Cap Tubes
LLC or CRI	1103	5	50-mL Screw Cap Tubes
ICSA	1104	6	50-mL Screw Cap Tubes
ICSAB	1105	7	50-mL Screw Cap Tubes
CCV	1106	8	50-mL Screw Cap Tubes
Samples	Racks 3, 4, & 5*	10+	50-mL Screw Cap Tubes
Samples	Rack 2*	10+	7 × 100−mm Tubes

*The Vial positions in each Rack are defined by a four digit number: the first digit is the Rack number, the second digit is the column number, and the last two digits are the row number. For example, the first tube in column 1 of Rack 1 is 1101; the first tube in column 1 of Rack 4 is 4101; etc. Rack 1 contains the run QC standards in 50-mL Screw Cap Tubes and the Vial positions are 1101-1107 (matrix D standards); 1201-1207(matrix H standards); & 1301-1307 (matrix F standards). Rack 2 is used for calibration standards, PDS, serial dilution, dilutions, and any other samples using 17 × 100−mm Tubes and Vial positions are 2101-2112; 2201-2212; 2301-2312; 2401-2412; & 2501-2512. Racks 3, 4 and 5 are used for samples in 50-mL digestion vessels in the Grey Foam Trays. Rack 3 uses Vial positions 3101-3105; 3201-3205; 3301-3305, 3401-3405 & 3501-3505. Rack 4 uses Vial positions 4101-4105; 4201-4205; 4301-4305, 4401-4405 & 4501-4505. Rack 5 uses Vial positions 5101-5105; 5201-5205; 5301-5305, 5401-5405& 5501-5505.There are also 10 Vial positions along the back of the autosampler that are designed for 50-mL Screw Cap Tubes, and these Vial positions are 1 through 10.

b. S0 and S1 must be poured fresh immediately prior to starting each analytical run. The check standards in Rack 1 are refilled as needed and must be kept capped when not in use.

c. Samples in the gray foam hotblock trays are placed directly on the autosampler using racks 3, 4, or 5.

d. For solid batches (10637, 5708) requiring dilution, samples must be poured into 17 × 100−mm Tubes and placed in Rack 2.

e. The sample log table used for analysis must be printed prior to commencing analysis. As each sample is placed on the autosampler, verify that the autosampler position on the table matches the placement of the tube.

NOTE: Any poured tubes must be labeled using the sample number, dilution factor and batch location; the pourer is responsible for making sure the samples are poured in the correct tubes and placed in the correct autosampler position.

9. Verify that all standards are the correct matrix and are in the correct autosampler tray position; they must not expire before they are analyzed in the run sequence.

10. Select *Sequence>> Run* from the ChemStation menu.

a. In the Start Sequence window type in your user ID (i.e., abc01234) as the operator name. Note that the default Data Batch Directory path is “C:\ICPCHEM\1 \DATA\[NAME].B\”, where [NAME] is the folder name assigned by the Agilent software.

b. The directory path must be changed to D:\ICPCHEM\1\DATA\[NAME].B\”. Verify that the data folder selected is unique from any previous runs.

c Once these changes have been made, click “Run Sequence”.

NOTE: To minimize downtime between analytical runs, it is acceptable to start the analytical run prior to pouring any of the samples. Setup the run as described above in Section D, pour the S0 and S1 and start the analytical run. Place the appropriate check standard cups in the autosampler, then proceed to pour the run using the printed autosampler table as a guide (record the appropriate information on the run cover sheet as you setup the standards and pour the run).

C. Operation of the Perkin-Elmer Elan 9000

1. To operate Elan software:

If not already active, double click on the Elan icon on the computer desktop to start the software.

2. Warm start-up of the Perkin-Elmer Elan 9000 ICP−MS:

a. Open the Instrument window and select the Front Panel tab.

b Verify that the System Status is “Ready”. If the System Status is “Not Ready”, click on the Diagnostics tab to find which parts of the system are not operating within specs. (See Section 14 of the Elan Version 2.4 Software Guide for further use of the diagnostics.)

c. Check that the waste drain vessel is not full, and has adequate space remaining to complete the automated run without overfilling.

d. Check that the sample, internal standard, and drain lines are attached to the peristaltic pump and that all tubing is connected properly.

e. Check to see that there is adequate internal standard solution to complete the entire automated run.

f. Check to see that the autosampler rinse vessel has sufficient rinse for duration of the automated run. If not, refill with the appropriate rinse solution.

g Both the sample and internal standard sipper probes must be placed in the 2−L bottle filled with reagent water.

h. Open the Instrument window and select the Front Panel tab. Check that the vacuum pressure reads between 1×10^{-6} Torr and 2×10^{-6} Torr. Pressure significantly lower or higher indicates a potential instrument hardware problem.

i Click on the “PLASMA START” button on the Front Panel tab. The plasma ignites within 20 seconds.

j After the plasma has ignited, open the Devices window and set the peristaltic pump speed to −24 rpm. If plasma fails to ignite, refer to Elan Software Guide section 2 “What to Do if Ignition Is Unsuccessful.”

k. Allow plasma to warm up for 10 to 15 minutes if system has been down for 15 minutes or less. Allow at least 30 minutes of warm up time if system had been down for more than 15 minutes.

3. Cold start-up of the Perkin-Elmer Elan 9000 ICP−MS

a. This procedure is used when the system has been down for an extended time period and the vacuum pumps have not been running.

b. Check oil levels in both vacuum pumps before proceeding.

c. Refer to Section 2 to 3 of the Elan Software manual for detailed instructions for starting up the instrument.

D. Perkin-Elmer Tuning and set up of Elan software

1. Open the Elan software if not already open.

NOTE: When the Elan software is first started, the analyst must always check to see that the tubing saver mode is disabled in the Devices window. The analyst must also verify that the correct sample details are entered in the Manual tab of the Sample window.

2. Daily Tuning.

a. After the instrument has been allowed to warm up, choose “Open Workspace” from the “File” menu and open “Daily Tuning.wrk.”

b. Aspirate the Elan 6100 Setup/Stab/Masscal Solution (10 ppb Mg, Cu, Rh, Cd, In, Ba, Ce, Pb, and U; before use each new bottle is also spiked with 0.2 mL of 1000 ppm Li for a concentration of 200 ppb Li). Be sure that both the sample probe and internal standard probe are placed into the solution. Allow the solution to flush until all air bubbles have traveled through both sample and internal standard lines. Cu, Ba, and Cd are not used in the evaluation of the instrument tuning.

c. Select the method and change the report filename using the 2 digit year, Julian Day, Tune number format (i.e., 0612301.TN#, where # is the instrument designation). Make sure the “Send to File” checkbox is checked; then save the method.

d. Select the Tuning window. Make sure the “Peak Width Only” box is checked, and click “TUNE MASS SPEC.” Five replicates of the Tuning solution are analyzed. Widths read back >= 0.64 and <= 0.66 amu. Verify that the Measured Mass is within ±0.1 amu of the Exact Mass. Check the Summary section and verify that the RSD is less than 5 for masses 6, 24, 103, 115, 140, 208, and 238. The Daily Tuning check is considered acceptable only when the Peak Width, Measured Mass, and RSD meet the specified criteria.

NOTE: RSD is calculated using 5 replicates.

NOTE: The Measured Mass values only change when a Mass Calibration is performed (i.e., tuning is performed with the “Peak Width Only” box unchecked), usually as part of optimization procedures. Thus, the Measured Mass values are usually already within the specified range. If not, perform a Mass Calibration (refer to the Elan version 2.4 Software Guide, p. 3−16). A Mass Calibration is only performed after any instrument maintenance issues have been addressed.

e. If peak widths fall outside of the specified range, they are adjusted by changing the RDAC Value. To lower the measured peak width by 0.01, raise the RDAC Value by 3 and vice versa. The new RDAC values are also determined by using the Excel spreadsheet “Tuning Calculator” located in K:\SHRDATA\ICP-MS. Separate spreadsheets are designated for each of the ICP-MS instruments in use.

Enter the Measured Peak Width values from the Tuning window into the “Measured Peak Width” column in the Excel spreadsheet. Enter the values from the “New DAC Value” column (in the Excel spreadsheet) into the appropriate RDAC Value cell in the Tuning window. After changing the appropriate RDAC values, save the tuning file (select “File”→“Save”) and click “TUNE MASS SPEC.” Repeat this process until all widths read back between 0.64 and 0.66 amu. Always save the tuning file before proceeding to Daily Performance.

NOTE: See Figure 1 for acceptance criteria.

NOTE: This check is performed once at the beginning of each day **OR** if maintenance is performed this check must be done after maintenance, before starting any analytical runs (even if Tuning has been done earlier on the same day).

3. Once daily Tuning passes criteria, choose “Open Workspace” and open “Daily Performance.wrk.” Continue to aspirate the Elan 6100 Setup/Stab/Masscal Solution. Type “Daily Performance” in the Sample Name field and click “ANALYZE SAMPLE.”

NOTE: This check is performed once at the beginning of each day OR if maintenance is performed this check must be done after maintenance, before starting any analytical runs (even if Daily Performance has been checked earlier on the same day). The criteria listed in Attachment I are posted as guidelines regarding general instrument performance. Achieving these criteria is not a prerequisite for sample analysis; however, they serve as indicators of the need for instrument maintenance and optimization. Run and batch QC are used to verify that the instrument performance is acceptable for each analytical run.

4. After Tuning and Daily Performance checks are complete select the Sampling tab in the Method. Click the “PROBE” button. The “Autosampler Probe Control” window appears. Click the “GO TO RINSE” button, then click “OK.” This returns the autosampler arm to the rinse station. Verify that the pump for the instrument rinse is turned on. Place the internal standard probe in the internal standard solution and place the sample probe into the rinse station (through the autosampler arm). If an acceptable Tuning is not achieved refer to the appropriate Elan Software Guide and/or the Elan 9000 Hardware Guide for troubleshooting the problem.

5. Before each analytical run begins, print a copy of the most recent Daily Performance Report for that day. A copy of the Tuning report automatically prints with each run when the run is imported.

NOTE: The Tuning data file must be imported to the IDAT database in LIMS prior to importing data for the first analytical run.

To reprint the Daily Performance Report open “Daily Performance.wrk”, select the Dataset window, highlight the last row of data, and click the “Reprocess” button.

6. Chose “Open Workspace” from the “File” menu and open the appropriate workspace. Typically “~Main 1.wrk” is used for the first run of the day; “~Main 2.wrk” is used for the second run of the day; etc.

a. Select the Method window and open an appropriate method.

b. Select the Sampling tab and change the rinse times to an appropriate value.

c. Click the Report tab on the right hand side of the Method window.

d. Under the section titled “Report to File”, change the Report Filename to the appropriate IDAT run # (i.e., c:\import\0612301.E01). The file name must end in “.E0#”, where # is the instrument designation.

e. Select “Append” then save the Method using “File”→“Save”.

f. Print the method file using the “MethodReport.rop” report options file.

7. Select the Sample window.

a. Open the appropriate sequence from the IDAT Sequences folder. All necessary fields are pre-filled by the Sequence Editor program at the time the sequence is created in LIMS.

b. “Wash (sec)” (i.e. rinse time) are adjusted depending on the sample matrix, but is the same throughout the entire run. A rinse time of 60 seconds is usually sufficient for most samples. If the run ends during your shift set “Wash Speed” value to −24 for the last tube in the run sequence. Optionally, a wash speed of −1 is used for the last tube in the run to reduce waste when the instrument is unattended.

c. When all information is verified in the Sample window save the file using "File"→"Save". Print the Sample file and double check all entries.

8. The autosampler positions (A/S) are defined as follows:

Standard ID	A/S	Sequence #	Tube type
S0 (Blank)	9	1	17 × 100−mm Tubes
S1 (Standard 1)	10	2	17 × 100−mm Tubes
CCS	2	4	50-mL Screw Cap Tubes
ICV	3	5	50-mL Screw Cap Tubes
ICB/CCB	4	6/11	50-mL Screw Cap Tubes
LLC or CRI	5	7	50-mL Screw Cap Tubes
ICSA	6	8	50-mL Screw Cap Tubes
ICSAB	7	9	50-mL Screw Cap Tubes
CCV	8	10	50-mL Screw Cap Tubes
Samples	11-157	12+	17 × 100−mm Tubes
Samples	158-182	12+	50-mL Screw Cap Tubes

a. S0 and S1 must be poured fresh immediately prior to starting each analytical run. The check standards in A/S 1-8 are refilled as needed and must be kept capped when not in use.

b. Samples begin at Sequence #12 and utilize A/S positions 158 to 182 when analyzed in the gray foam hotblock trays (containing up to 25 x 50mL digestion cups).

c. A/S positions 11 to 37 are used for any dilutions or PDSs (poured into 17 × 100−mm Tubes) when a hotblock tray is used.

d. For sample batches not using the hotblock trays, samples are poured into

e. The autosampler table used for analysis must always be printed prior to placing the samples on the autosampler. As each sample is placed, verify that the autosampler position on the table matches the placement of the tube in the sample racks (the numbered autosampler trays are used whenever possible).

9. Verify that all standards are the correct matrix, are in the correct autosampler tray position and have sufficient time until expiration to complete the run.

a. Open the Calibration View window and open a new calibration file using "File"→"New." If prompted to save the calibration file select "No." If prompted to clear blank and remove calibration information, always select "Yes."

b. Select the "Sample" window, save the sample file, and then save the workspace.

c. Highlight all samples to be run, and click "Analyze Batch." The system runs the Blank and calibration standard, then check standards and samples.

d. If prompted to clear QC data and/or all existing Blank/Calibration data, always select "Yes."

NOTE: To minimize downtime between analytical runs, it is acceptable to start the analytical run prior to placing any of the samples. Setup the run as described above in Section D, pour the S0 and S1 and start the analytical run. Place the appropriate check standard cups in the autosampler, then proceed to load the autosampler using the printed autosampler table as a guide (record the appropriate information on the run cover sheet as you setup the standards and pour the run).

NOTE: The instrument methods used for sample analysis are configured to take the average of three replicate readings of each standard and sample at the time of analysis. This average is calculated by the instrument software. The average result is used to calculate the final samples results.

NOTE: The instrument methods used for sample analysis are configured to use the "Linear Thru Zero" curve type. The Elan 3.0 software guide definition of "Linear Thru Zero" curve type is: "Uses linear regression with a forced zero"; i.e. the curve includes a point at the origin of the calibration graph.

E. Import, QC Review and Upload of the sample data using IDAT

1. After the analysis is complete the analyst must run a macro to create a CSV file and place it in the "D:\IDAT\Import\" directory. The file name matches the data directory for each run.

a. In the Offline Data Analysis program open the appropriate data file, method and calibration for the run you are importing. Verify in the drop down menus that the data directory matches the raw data printout.

b. Select “DoList” from the “Tools” menu. Verify that “Macro” is the only option checked in the “Select a Report Option” window, then click “Go”.

c. Chose “a_a_Data.mac” from the list of macro files and click “OK”.

d. Select the calibration blank through the last CCB in the list of files, click “Add” to move them to the right, and then click “Process”. The screen flashes as the macro processes the data. “File list processed” appears in the lower left corner when the macro is finished.

2. Open LIMS and Login. Select *IDAT>> Import*.

3. Double−click on the CSV file that corresponds to the run you are importing.

4. Verify that all information is correct. Report any missing or incorrect prep data to your supervisor and make a note in the comments section of the batch sheet.

5. Enter the employee number for the analyst listed as Analyst at the top of the run cover sheet. DO NOT use your own employee number unless you are the Analyst for the run.

6. Enter the rinse time; then click “Save”.

7. Once import completes, verify that the run log is filled out correctly. Click OK to exit the import program.

8. Select *IDAT>> QC Review*. Select the appropriate run number from the list then click “Review”.

9. Once the review is complete, press the “Print QC” button to get a copy of the QC review. This is not necessary if you have “Print QC Report” checked.

10. View the list of samples that did not pass by selecting the radio button next to “Bad Samples”. The “Yttrium” tab also shows internal standard recoveries. These two tools are used in conjunction with viewing the raw data printout and IDAT run report for each sample in order to determine appropriate dilution factors for any samples requiring rereads (either due to analytes over the linear range, the presence of internal standards in the sample, or matrix effects which causes internal standard drift). Print the Reread report, if needed.

11. Note any dilutions on the Reread Report, then sign and date the top of the reread report.

12 Close QC review once review of the data is complete. Select *IDAT >> Upload*.

13. Click “New Runs”; then select the appropriate run number and click “Upload”. Click “OK”; then close Upload.

F. Operation of the Agilent 7700 ICP-MS

NOTE: Additional information on the operation of the MassHunter Workstation software can be found in the Agilent 7700 Series ICP-MS Masshunter Workstation User Guide.

1. Startup of the Agilent 7700 ICP-MS

a. Power on the ESI autosampler and start the ESI software.

b. Make sure the instrument is powered on before starting the ICP-MS software – the light on the front of the instrument will be red (shutdown mode), orange (standby mode) or green (analysis mode).

c. If no light on the front of the instrument and the instrument is powered down: flip the main power switch on the back of the instrument up to the on position, and then make sure the Foreline Pump (red switch) is also up in the on position.

d. Make sure the cool flow power switch is up in the on position. If the orange light on the front of the cool flow is on, you will need to turn off the cool flow and add coolant then turn the cool flow back on.

CAUTION: Before turning off the cool flow the instrument MUST be in standby, shutdown or powered off – DO NOT turn off the cool flow with the plasma on.

e. Start ICP-MS Instrument Control (Masshunter).

f. Select Instrument Control upon program startup.

g. In lower left corner there are 4 buttons: Startup, Batch, Queue, and Hardware.

h. Select Hardware.

i. Verify that the program is in Online mode – the title bar should read “Online ICP-MS Mass Hunter”.

j. If the title bar says “Offline ICP-MS Mass Hunter” right click on the Mainframe picture in the Hardware pane and select Communication. Click the tick mark next to Online and click OK.

k. Check the Instrument Status pane. The instrument should be in one of three modes: Shutdown (plasma and vacuum off, red light on front of instrument); Standby (plasma off but vacuum on, orange light on front of instrument); Analysis (plasma and vacuum on, green light on front of instrument).

l. If in Shutdown mode right click on the Mainframe picture and select Vacuum On – the Foreline pump will start and the instrument will transition to Standby mode.

m. Once Standby mode has been reached select Plasma On from either the Instrument drop down menu or the button at the top center of the software window.

n. When asked if you want to “Run ‘Startup’ after plasma ignition?”, select No.

o. The plasma will ignite and the instrument will transition to Analysis mode.

p. Once Analysis mode is achieved you must allow the instrument to warm up for at least 20 minutes before running the EPA Daily Tune check sample.

2. Running the Daily Tune Check on the Agilent 7700 ICP-MS

a. To run the EPA Daily Tune check place both probes into the EPA Tune Solution and allow sufficient time for the solution to flush through both lines (the internal standard line will take longer to flush).

b. Make sure to uncheck “FAST Control Enabled” in the ESI software and ensure that the FAST valve is in the LOAD position with the FAST vacuum off. Ensure that the cap for the sample cup in position 1 has been removed.

c. Once the EPA Tune Solution is aspirating into the spray chamber take the following steps to start the EPA Daily Tune check.

d. Select the Batch button in the lower left corner of the ICP-MS software.

e. Open the batch “EPATUNE.b” by clicking on the following folder:

D:\Agilent\ICPMH\1\DATA\~EPATUNE.b

f. In the Batch pane select the Acq Method tab then the Tune tab.

g. Right click in the Signal Monitor grid and select Report, then Generate Tune Report.

h. In the window that appears verify that the Report Template selected is as follows:

C:\Agilent\ICPMH\Report Templates\en\Letter\Tune Report\TuneCheckReport.xltx

i. Click Generate. The software will acquire the tune check data from the instrument and a report will be printed.

j. If the EPA Daily Tune fails any of the criteria rerun the tune a second time. If it fails a second time take corrective action before running the EPA Daily Tune check again (maintenance or optimization of tuning parameters).

3. Sample analysis on the Agilent 7700 ICP-MS

a. Once the EPA Daily Tune passes all criteria, place the internal standard probe in the internal standard solution and place the carrier probe into the carrier solution.

b. Once the carrier and internal standard solution have flushed through the tubing and are aspirating into the spray chamber, sample analysis can begin.

c. Open a batch folder that is suitable for the type of samples and analytes to be analyzed (Example: “~ICPMS.b” when all three gas modes are needed).

d. Save the batch with a new name using the same convention that is used in the Chemstation software on the Agilent 7500 ICP-MS:

YYMDDR## where

YY is the two digit year,

M is the letter of the alphabet corresponding to the month (A for January, B for February, etc.)

DD is the two digit day of the month

R is the letter of the alphabet that changes for each attempted run sequence

Example: For the first run started on March 19 in the year 2012 the batch folder would be named 12C19A01.b

NOTE: If you need to remove a run and start another run you can also advance the 01 to 02 etc. to create a unique batch for each run.

e. Select the Sample List tab in the Batch pane.

f. Under Sequence Flow the Blank Samples block may be used for running pre run rinses and conditioning samples (clear this table if you don't need pre run rinses or conditioning, or adjust this table as needed to condition the instrument for your run).

g. The Unknown Sample block is used to run the sample sequence.

h. Select the Unknown Sample block and clear the sample table, then right click on the first row in the sample table and select Import Sample List.

i. Select the appropriate CSV file for the sample batch you will be analyzing and the sequence table will be filled in.

j. Verify the entries are correct and match the samples and locations for the samples to be analyzed and load calibration standards, check standards and samples into the correct positions on the autosampler.

k. Click Add to Queue to add the current Sample List to the analysis and to begin the run – if it is the first run you are adding to the Queue the autosampler will move to the first position in the Blank Sample block right after you click Add to Queue.

l. Once the Queue is active and running samples you can repeat the above steps to add additional runs to the Queue.

4. Data analysis and import for the Agilent 7700 ICP-MS:

a. Once the sample run is complete you will switch to or open the Offline ICP-MS Data Analysis program to make any changes to internal standard selection and generate the file for import into LIMS.

b. Once in the Offline ICP-MS Data Analysis program open the batch that contains the run data you are going to process by clicking Open Analysis File (open folder icon or from the File drop down menu).

c. Evaluate appropriate internal standard selection based on the Quality Control section of this SOP and method requirements for the samples analyzed.

d. If it is appropriate to change internal standard selection from the defaults in the method used click the DA Method Editor button (skip to step e. if alternate internal standard selection is not needed).

(1) Under Method Development Tasks (in the left pane of the DA Method Editor window) select “FullQuant” under item number 4 (Set up Analysis Parameters).

(2) Under the analyte list that appears in the right hand pane of the DA Method Editor you will see a list of the analytes.

(3) Change internal standards for any analytes where alternate internal standard selection is both necessary and appropriate.

(4) Then click “Return to Batch-at-a-Glance” in the left hand pane.

(5) When prompted to “Update Analysis Method?” select “Yes”.

(6) Click the “Process Batch” button to recalculate all concentration values in the batch folder using the internal standard selections you made while in the DA Method Editor.

e. To generate the file for import into LIMS you must first select all the data rows needed for your run, starting with the calib blank down to the last row in your run. If you restarted the run only select the most recent calib blank and all samples below it.

f. Once the appropriate rows of data are selected click Actions from the Tools drop down menu and select “Generate Import File” to run the script that compiles the file for import.

g. Once the script is complete you will get a message indicating the location and name for the import file (Example “File Location: C:\IDAT\Import\12H14B01.E05”).

h. Click OK then open LIMS and Import the data file.

Routine Maintenance

A. Maintenance for the Agilent 7500 ICP-MS and ESI SC-8 FAST Autosampler

1. Documentation

Any adjustment to an instrument, replacement of parts, etc., must be documented in the appropriate instrument logbook.

NOTE: For more details on procedures in this document please refer to the Agilent 7500 Hardware Manual or the Agilent 7500 Series ICP-MS Maintenance Video (DVD). The Agilent 7500 Series ICP-MS Maintenance Video contains high quality video and complete descriptions of all necessary maintenance tasks.

NOTE: Since the system is now using the ESI SC-8 FAST autosampler, ESI PC^3 cooled spray chamber, and ESI nebulizer, some components of the sample introduction system are different from those described in the Agilent 7500 Series ICP-MS Maintenance Video.

2. Routine maintenance

a. Remove and clean the sample introduction system when instrument performance declines.

(1) Clean nebulizer and spray chamber.

(a) Put in standby mode (Turn off plasma in ChemStation).

(b) Flip up dust shield. Open top cover.

(c) Remove torch box with two bolts.

(d) Remove two metal clip clamps.

(e) Disconnect the makeup gas line and remove glass sample transfer tube.

(f) Unthread the mixing tee from the end of the nebulizer, then unthread and remove the nebulizer from the spray chamber. Leave the gas tubing attached to the ESI nebulizer and detach it from the top of the Argon humidifier instead.

(g) Remove chiller top cover by loosening the white bolt.

(h) Lift spray chamber and carefully remove drain line.

(i) Unthread the plastic cap that is attached to the optional gas addition port on the top of the spray chamber and set aside.

(j) Pump clean water through nebulizer in the opposite direction. Be very careful not to handle the nebulizer more than necessary to avoid damage to the tip.

(k) Sonicate spray chamber and glass transfer tube in 10% nitric. Rinse with reagent water.

(l) Dry the outside of the spray chamber and transfer tube with a paper towel. Remove any excess water from the spray chamber using compressed air. It is not necessary to completely dry the inside of the spray chamber.

(m) The transfer tube must be completely dry before reinstallation. Accelerate drying, if necessary, placing the transfer tube in a drying oven set to 100 degrees Celsius for approximately 10 minutes.

(2) Removing and cleaning the torch

(a) Follow steps (1)i through (1)v in nebulizer removal section above.

(b) Remove gas tubings from torch. Inspect the gas tubing lines: notify a supervisor immediately if any cracks or signs of weakness are observed.

(c) Open quick release clamp. Remove from coil.

(d) Remove bonnet and shield bar (the glass sleeve and thin metal sleeve).

(e) Check bonnet and shield bar for cracks or deformation.

(f) Sonicate torch and bonnet in 10% nitric. Rinse with reagent water.

(g) Replace torch if it shows excessive wear, or build−up that cannot be removed.

(h) The torch and bonnet must be completely dry before reinstallation. Drying may be accelerated by placing these parts in a drying oven set to 100 degrees Celsius for approximately 10 minutes.

(3) Reassembling sample introduction system

(a) Put shield bar and bonnet back on torch.

(b) Slide torch into coil. Make sure the small glass bump is aligned with the corresponding hole on the torch stand to ensure proper torch alignment.

(c) Close quick release clamp.

(d) Reconnect gas lines. Gas lines are different sizes to avoid accidental incorrect connection.

(e) Reassemble spray chamber. Attach drain line and put spray chamber back in chiller. Replace top cover and tighten plastic bolt.

(f) Reattach the nebulizer gas line to the top of the humidifier. Rethread the mixing tee to the end of the nebulizer. Reattach the plastic cap on the optional gas port on top of the spray chamber.

(g) Replace glass sample transfer line. Reattach with metal clips and reattach makeup gas line.

(h) Replace torch box and tighten it down. Close top cover.

(4) Cleaning the FAST valve assembly

NOTE: Detailed descriptions and diagrams of the FAST valve assembly are located in the pdf document “ESI SC Manual.pdf” located in the dept. 22 folder at K:\DEPT22\ICPMS Training and Reference Materials.

(a) Periodically clean the FAST valve assembly when instrument performance declines and instrument maintenance does not correct the problem.

(b) In the ChemStation software open the tuning window and stop the nebulizer pump.

(c) Unthread and set aside all six connections going into the FAST valve head.

(d) Use a 9/64” Allen wrench to remove the valve head from the FAST hut.

(e) Use a 7/64” Allen wrench to remove the three screws from the valve assembly.

(f) Disassemble the valve assembly and place components on a clean dry surface.

(g) Rinse the rotor with reagent water and use compressed air to remove any deposits. Remove stubborn deposits by sonicating the rotor in a solution of up to 10% nitric acid for approximately 10 minutes, followed by rinsing with reagent water. Replace the rotor if it shows signs of excessive wear. Dry rotor with compressed air prior to reinstallation.

(h) Use compressed air on each of the six ports on the stator. Visually inspect the openings and confirm that nothing obstructs any of the six ports.

(i) Reassemble the valve assembly. When reinserting the 3 screws, alternate between the 3 screws to keep them evenly threaded.

(j) It is very important to avoid over tightening the 3 screws. Once you reach the point where the stator, body ring, and valve body are flush with one another tighten each screw gently until it feels snug (using forefinger and thumb only). DO NOT tighten with full force to avoid damage to the valve assembly.

(k) Reattach the valve assembly to the FAST hut. While keeping the valve assembly firmly seated against the hut, use the 9/64” Allen wrench to firmly tighten the screw. It is necessary to apply enough force to make sure the

valve head does not rotate when the FAST switches between LOAD and INJECT.

(l) Carefully thread each of the six connections to the valve head. You MUST avoid cross threading and over tightening to avoid damaging the threaded connections. Threading must be smooth. If you encounter resistance STOP, reverse and restart the threading. Gently finger-tighten each connection.

b. Removing and cleaning the cones.

(1) Removing cones

(a) Follow steps i. through v. in “Cleaning Nebulizer” section above

(b) On the ChemStation instrument control panel click maintenance and then choose sample introduction. Click the maintenance button in the window. This moves the torch housing and nebulizer out and away from the cones.

NOTE: Move the argon humidifier aside so that the FAST valve assembly does not collide with it as the torch housing moves.

(c) Use sampling cone wrench line up so that the pins fit in the holes on the outer part of the cone. Place a long screwdriver through the holes on the wrench for leverage in loosening the sample cone.

(d) Slide skimmer cone wrench into hole where skimmer cone is and turn until magnets lock pins into place. Use screwdriver for leverage in loosening the skimmer cone.

(2) Cleaning cones

(a) Remove the O-ring from the larger sampling cone and rinse cones in reagent water. Use a wet paper towel to help remove deposits.

(b) If deposits are still observed after completing step i., additional cleaning must be performed following steps iii. and iv. below. Otherwise skip to step v. below.

(c) Prepare a slurry using a small amount of alumina powder and reagent water. Dip a cotton tipped swab in the slurry and gently polish the inner and outer surfaces of the cone. To clean the cone orifice, the cotton swab is inserted in the back of the cone using a twisting motion to remove deposits. DO NOT polish any threaded portion of either cone with the slurry.

(d) More aggressive deposits are removed using #400 grit waterproof abrasive paper. Gently polish the cone surfaces with the cone still wet. DO NOT polish the tip of either cone or the threaded portions of either cone.

(e) Rinse each cone thoroughly with reagent water to remove all traces of loose deposits and alumina powder.

(f) Sonicate cones in a beaker containing only reagent water for 20 minutes.

(g) Remove form Beaker. Rinse with reagent water and dry. Accelerate drying, if necessary, by placing the cones in an oven set to 100°C for approximately 10 minutes. Cones must be completely dry before reinstallation.

(h) Visually check the tip of the cone and the back of the cone for abnormalities. Replace any cone that appears to have an oversized or non−round tip, or has surface deformities on the back of the cone.

(i) Visually inspect O-ring for cracks or hard spots. Replace if necessary.

(3) Reinstalling cones

(a) Put skimmer cone in the skimmer cone wrench and turn until magnets lock pins into place. Slide wrench with cone attached into cone housing and carefully thread the cone back into place. Be sure to not cross thread. Tighten this cone down by using the screwdriver for leverage.

(b) Carefully thread the sampler cone back into housing by hand. Be careful not to cross thread it. Before tightening with the cone wrench refer to the maintenance video titled “Installing the Sampling Cone” and follow the procedure exactly to obtain the correct tightness: first hand tighten without the O-ring in place; then mark the cone and interface; next, remove the cone and reinstall with the O−ring in place; tighten using the cone wrench and

screwdriver for additional leverage so the marks on the cone and interface match. DO NOT tighten past the mark to avoid damage to the cone and/or interface.

(c) In ChemStation, go to “maintenance” on the instrument panel and select sample introduction. Click the initialize button. This moves the torch housing and nebulizer forward.

(d) Replace the torch box.

(e) Close cover.

c. Removing and cleaning the lenses

(1) Removing Lenses (There are multiple lenses to clean: the Extraction Lens-Omega-Lens assembly, the Reaction cell which contains the Cell Focus and Cell Entrance Lens, Plate Bias Lens.)

(a) Follow steps i. through v. in the Cleaning Nebulizer section.

(b) Click “maintenance” in the ChemStation instrument control panel and choose sample introduction. Click the “maintenance” button in the window. This moves the torch housing and nebulizer out and away from the cones.

NOTE: If you are not cleaning the Reaction Cell it is not necessary to put the instrument in shutdown mode or to vent the vacuum chamber. Once the instrument is in standby mode skip to step vii. below. DO NOT put the instrument in shutdown mode or vent the vacuum chamber if you are only cleaning the Extraction Lens-Omega-Lens assembly.

(c) Put the instrument in shutdown mode (turn off vacuum). Turn computer off and then turn off instrument using the button on the lower right of the front of the instrument.

(d) Flip open cover. Turn the vent valve (located on the front left of the vacuum chamber) one turn. A slight hissing sound is heard.

(e) While the chamber is hissing remove screws on the vacuum chamber lid.

(f) Once the vacuum chamber has equalized, remove the lid. Remember to retighten the vent valve. Rest the lid on top when not working in the vacuum chamber to avoid entrance of dust and particles.

(g) Remove the sampling cone and skimmer cone by following steps iii. and iv. in Removing cones section.

(h) Loosen three screws on skimmer base. Remove Extraction Lens-Omega Lens assembly. Carefully remove three screws on the Omega Bias. Slide off each lens and keep spacers in order. There are four lenses.

NOTE: If you are not cleaning the Reaction Cell skip to Cleaning lenses section (2) below. Follow steps ix. through xii. only if you are cleaning the Reaction Cell.

(i) Open vacuum lid and loosen reaction cell screw. Loosen but do not completely remove plate bias wire terminal screw. Remove wire. Unplug plastic wire connector. Carefully remove reaction cell by lifting out at an angle.

(j) Loosen but do not completely remove plate bias wire screws. Remove wire ends. Remove the four cell assembly screws. Separate housing and carefully remove octopole unit. Be sure not to touch the octopole to avoid misaligning the poles.

(k) Remove the two screws from the entrance side of the octopole. Slide off the cell focus, spacer and cell entrance lens. Secure octopole plate with same screws. Remove the other two screws from the exit side. Remove cell exit lens, spacer and QP focus. Secure octopole plate with same screws. Place octopole unit aside to avoid damage. There are four lenses.

l) Open the vacuum chamber lid and loosen the plate bias lens screws. Remove cell exit guide and plate bias lens.

(2) Cleaning lenses

(a) Polish each lens with #400 polishing paper. Then use #1200 paper. Clean the orifice of each lens using an alumina powder slurry.

(b) Rinse with water.

(c) Sonicate in water for 5 minutes after polishing.

(d) Rinse with water.

(e) Sonicate in water for additional five minutes.

(f) Allow lenses to dry. Do not wipe. Accelerate drying, if necessary, by placing the lenses in an oven set to 100 degrees Celsius for approximately 10 minutes. Lenses must be completely dry before reinstallation.

(3) Reinstalling lenses

NOTE: If you are not cleaning the Reaction Cell skip to step vi. below. Follow steps i. through v. only if you are cleaning the Reaction Cell.

(a) Remove vacuum chamber cover. Put plate bias lens and cell exit guide in place. Secure with screws.

(b) Remove each octopole plate and reattach the lenses on either side of the octopole. Be aware that there are two different sized holes so that the lenses go back on in the correct orientation. Place reassembled octopole unit into the reaction cell and put the four screws back in.

(c) Reattach the wires to the back of the reaction cell. The top screw holds down the wire that is attached to the reaction cell. The bottom screw holds the currently loose wire.

(d) Carefully put the reaction cell back into the vacuum chamber. Tighten the reaction cell screw. Replace wire in the plate bias lens screw and tighten screw. Plug the wire connector back in.

(e) Put the vacuum chamber lid back on. Secure it with the four screws tightly. Make sure the vent valve is tight as well. Close top cover.

(f) Reassemble the entrance lens-omega lens. Slide into skimmer housing. Tighten the screws.

(g) Put cones back in. Use steps i. and ii. in reinstalling cones section.

(h) Turn instrument and computer on. Turn on vacuum in ChemStation. Wait 15 to 60 minutes for the vacuum to pump down. Once instrument is pumped down, go into the Maintenance menu and then sample introduction. Click the initialize button. This moves the torch housing back into place. Reattach torch box and secure with screws. Close top cover.

(i) Turn Plasma on. Give the instrument time to equilibrate with the vacuum and the plasma both on. Check instrument performance.

d. Replace pump tubing when it shows stretching, flat spots, or increased relative standard deviations. Inspect all tubing to insure that it is secure and in good condition. Periodically perform visual check on the oil level of vacuum pumps and the water level in the coolflow. Also check water in coolflow for algae growth.

B. Maintenance for the Perkin Elmer Elan 9000 ICP-MS

For more details on procedures in this document (including detailed pictures) refer to the Elan 9000 Hardware Guide.

NOTE: Any adjustment to an instrument, replacement of parts, etc., must be documented in the appropriate instrument logbook.

1. Routine Maintenance

a. Remove and clean the sample introduction system when instrument performance declines.

(1) Removing spray chamber

(a) Remove dust cover located above spray chamber

(b) Disconnect sample capillary tubing and argon gas tubing from nebulizer.

(c) Unscrew spray chamber retaining ring completely (plastic–knurled ring) and remove spray chamber from torch assembly.

(d) Loosen two knurled screws and remove nebulizer from spray chamber.

(e) To clean spray chamber, remove drain tubing and rinse with reagent water.

(f) Clean nebulizer by sonicating in 10% HNO_3 for a few minutes.

(2) Removing and cleaning the torch

(a) Use Allen wrench to push release mechanism on interface lever. Open interface by moving lever fully to the right.

(b) Remove torch−box cowling by loosening the two black−knurled knobs. Set aside. (**Caution:** there is an electrical connection; cowling cannot be removed, just move out of the way.)

(c) Remove gas line connections from the torch by loosening the Swagelock fittings a few turns. Then slide the fittings and tubing up and away from the torch.

(d) Rotate the torch mount (large−knurled metal ring) an eighth of a turn counterclockwise, and remove entire torch assembly from spray chamber side.

(e) Separate the torch from the adapter. Pull torch tip out of the adapter.

(f) Sonicate torch in 10% HNO_3 for a few minutes. Rinse with reagent water.

(g) Inspect tip. Sonicate along with torch if necessary. Rinse with reagent water.

(h) Replace torch and/or tip if either shows excessive wear, or build−up that cannot be removed.

(3) Reassembling sample introduction system

(a) Slide tip back into adapter. Next slide torch onto adapter. Both pieces must slide in until they hit a stop.

(b) Feed torch through hole on right side of instrument. Rotate torch assembly an eighth of a turn clockwise until bayonet mount is fully seated.

(c) Slide both gas fittings and tubing onto torch ends. Fittings are marked “B” for the back fitting and “F” for the front fitting. Tighten finger tight.

(d) Reinstall torch box cowling. Both black−knurled knobs must be snug, but not over tightened.

(e) Place torch alignment tool on torch so that it butts up against the outermost RF coil.

(f) Loosen the torch−locking collar (smaller−metal knurled ring) until torch moves freely. Then align the torch so that it is even with the outermost edge of the alignment tool. Retighten torch−locking collar.

(g) Slide tool out approx. ½ inch. Then close the interface by moving the lever full to the left.

(h) Open the interface by moving the lever fully to the right. The edge of the torch must be aligned with the 5.5 mm cutout of the alignment tool. (If not, refer to Elan 9000 hardware guide)

(i) Remove alignment tool.

(j) Close interface by moving lever fully to the left. (Safety interlock must click into place.)

(k) Reconnect nebulizer to spray chamber. Tighten both knurled screws finger tight. Align nebulizer so that the sample capillary tubing inlet is at approx. the 2 o’clock position when installed on the torch.

(l) Reconnect argon gas, sample capillary, and drain tube fittings.

(m) Reconnect spray chamber to torch extension with back end of chamber angled down for proper drainage.

(n) Finger tighten spray chamber retaining ring. Recheck torch alignment.

(o) Optimize the procedures in Section 3 of the Elan software guide.

b. Removing and cleaning the cones

(1) Removal of cones

(a) Turn off plasma if plasma is lit. Allow two (2) minutes or more for cooling and vacuum to equilibrate.

(b) Open top cover. Use Allen wrench to push release mechanism on interface lever. Open interface by moving lever fully to the right.

(c) Align removal/insertion tool with cutout in sampler cone. Insert and rotate a quarter turn. Pull cone forward using rocking twisting motion.

(d) Reverse removal/insertion tool so pins face skimmer cone. Seat tool into holes in cone. Turn tool counterclockwise to remove cone.

(2) Cleaning cones

(a) Remove O-rings from both cones and rinse cones in reagent water. A wet paper towel may also be used to help remove deposits.

(b) If deposits are still observed after completing step i. ; additional cleaning must be performed following steps iii. and iv., otherwise skip to step v..

(c) Prepare a slurry using a small amount of alumina powder and DI water. Dip a cotton tipped swab in the slurry and gently polish the inner and outer surfaces of the cone. To clean the cone orifice, the cotton swab may be inserted in the back of the cone using a twisting motion to remove deposits. DO NOT polish the threaded portion of the skimmer cone or the outside edge of the sampler cone with the slurry.

(d) More aggressive deposits may be removed using #400 grit waterproof abrasive paper. Gently polish the cone surfaces with the cone still wet. DO NOT polish the tip of either cone, the outside edge of the sampler cone or the threaded portion of the skimmer cone.

(e) Rinse each cone thoroughly with reagent water to remove all traces of loose deposits and alumina powder.

(f) Sonicate cones in a beaker containing only DI water for 20 minutes.

(g) Remove from beaker. Rinse with reagent water and dry.

(h) Visually check tip of cone for abnormalities. Replace any cone that appears to have an oversized or non−round tip.

(i) Visually inspect O-rings for cracks or hard spots. Replace if necessary.

(3) Reinstalling cones

(a) Using removal/insertion tool, screw skimmer cone clockwise into place.

(b) Reverse tool and install sampler cone. Cone is pressure fitted into place. Cone seats flush with interface.

(c) Move interface region back into operating mode by moving interface lever fully to the left.

(d) Restart plasma. Sampler cone will fully seat when vacuum pump starts.

(e) Optimize following procedures in Section 3 of the Elan software guide.

c. Removing and cleaning the lens

(1) Removing the Lens

(a) Shut down plasma if lit and shut down vacuum pumps.

(b) Wait at least 15 minutes for vacuum chamber to vent.

(c) Using a Phillips screwdriver, loosen screws that hold ion optics region vacuum chamber lid in place (right-hand lid, smaller of the two). Remove lid. (May require a little prying to break seal, use caution).

(d) Detach electrical connection from lens. This is the gray ball and socket connector on top.

(e) Using 2 mm Allen wrench, loosen the two lens mounting screws. DO NOT REMOVE SCREWS.

(f) Remove lens by rotating it slightly counter clockwise and pulling it out. Remove the shadow stop by rotating counter clockwise and pulling it out.

(g) Unscrew lens mount nut and remove the small insulator. Slide the lens out of the mount.

(2) Cleaning the lens and shadow stop

(a) Place lens in a beaker containing no more than 2.5% HNO_3.

(b) Sonicate for no more than 5 minutes. DO NOT leave the lens in the acid for more than 5 minutes.

(c) Remove lens from beaker and rinse with reagent water, methanol then reagent water again.

(d) Blow dry. Reinstall.

(e) If unable to sufficiently remove residue, replace with new lens.

(f) Shadow stop must be cleaned by wiping off surface with 2.5% HNO_3 on a cotton swab. Sonicate with lens if needed. Rinse same as lens.

(3) Reinstalling Lens

(a) Place lens into lens mount.

(b) Place small insulator on top of lens.

(c) Tighten the lens–mount nut back into place. Finger tight is sufficient.

(d) Place shadow stop onto mounting screw and rotate clockwise to lock. Next place lens assembly onto mounting screws and rotate clockwise to lock. Make sure electrical connector is facing up.

(e) Tighten screws.

(f) Reattach electrical connector.

(g) Replace vacuum chamber lid, making sure cut-out is back and to the right. Tighten screws. (Do not over tighten)

(h) Restart vacuum by pressing switch on the left side of the instrument, or through the Vacuum Start button in the Instrument window of the software.

(i) Once system reaches adequate vacuum and the system comes to ready, retighten screws on vacuum chamber lid.

(j) Optimize the following procedures in Section 3 of the Elan software guide.

d. Replace pump tubing when it shows stretching, flat spots, or RSDs start drifting up. Inspect all tubing to insure that it is secure and in good condition. Periodically perform visual check on the oil level of vacuum pumps and the water level in the coolflow.

C. Preventative Maintenance (performed as needed) for the Agilent 7500, Agilent 7700 and Perkin Elmer Elan 9000.

1. Vacuum instrument air filters

2. Vacuum coolflow air filters; coolflow air filters may also be removed and run under tap water to remove dust build up.

3. Change vacuum pump oil

4. Replace interface rough pump oil

5. Replace turbo backing pump oil

D. Nonroutine Maintenance for both Agilent 7500 and Perkin Elmer Elan 9000

Sections 4 and 5 of both the Agilent 7500 Hardware Manual and the Perkin Elmer Elan 9000 Hardware Guide contain more information on the maintenance and trouble shooting of the ICP−MS. Consult an Agilent or Perkin Elmer service representative for further information on troubleshooting and maintenance.

E. Taking an instrument/analysis out of service/returning an instrument/analysis to service

NOTE: The following information is taken from 1−P−QM−QMA−9017325. In the event of an equipment failure, perform the following:

1. Document the nature of the failure in the maintenance logbook.

2. Document how and when the defect was discovered.

3. Notify a supervisor or responsible person to decide on appropriate action to take.

4. The instrument must be clearly tagged as *Out of Service*. The tag must contain the following information:

    a. Date taken out of service

    b. Employee who took the instrument out of service

    c. Reason for tagout

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken to bring the equipment back into service.

7. Document results of the corrective action (e.g., system calibration within specifications, etc.)

8. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact of the failure on the calibration and/or qualification of the instrument.

9. After repair, document whether the function has been fixed. Calibration or verification activities are performed before the instrumentation is put back into service.

F. Maintenance for the Agilent 7700 ICP-MS

For detailed instructions for all maintenance refer to the Agilent 7700 Series ICP-MS Hardware Maintenance Manual.

Calculations

1. Final Result

a. Water sample

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

All dilution factors must be recorded and used in the calculation. [To enter dilution data into the LIMS when multiple dilutions are used, a factor must be formed (Ex. 1), which contains no more than three figures for the volume or the aliquot (Ex. 2).]

Ex. 1. 50/.5 × 10/1 = 500/.5

Ex. 2. 50/.5 × 25/.5 = 1250/.25 = 125/.025

NOTE: The default units are µg/L

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{N}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{N}\right)}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\%\ Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)
Take either 4 identical aliquots (for 3 point MSA) or 2 identical aliquots (for one point MSA) of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution (for 3 point MSA) and spike the other aliquot at approximately the indigenous concentration of the sample (for one point MSA). Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots or 2 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^2 - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{\left[\sum (x_i - \bar{x})^2\right]\left[\sum (y_i - \bar{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ≥0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

7. Average, Standard Deviation and Relative Standard Deviation

The average result, $\bar{x}$, is calculated by summing the individual results and dividing this sum by the number (n) of individual values:

$$\bar{x} = \frac{x_1 + x_2 + x_3 + x_4 + \ldots x_n}{n}$$

Standard deviation is a measure of how precise the average is or how well the individual numbers agree with each other.

$$\text{Standard deviation, } S = \sqrt{\frac{(x_1 - \bar{x})^2 + (x_2 - \bar{x})^2 + (x_3 - \bar{x})^2 + \ldots + (x_n - \bar{x})^2}{n-1}}$$

Relative standard deviation (RSD) is expressed in percent and is obtained by multiplying the standard deviation by 100 and dividing this product by the average.

$$\text{Relative standard deviation, RSD} = \frac{100 \times S}{\bar{x}}$$

Statistical Information/Method Performance

Generate MDLs and LOQs according to 1–P–QM–QMA-9017309. Perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire digestion and analysis procedure. Compare and pool results to determine the final reporting MDL. The department supervisor maintains annual study data. The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

Refer to 1−P−QM−QMA−9017313 for statistical information.

Quality Assurance/Quality Control

A. For 6020, 6020A and 6020B, each digestion batch (up to 20 samples) must contain a method blank, LCS, and either an US, D, MS, MSD or an LCS/LCSD.

B. For 200.8, each digestion batch (up to 10 samples) must contain a method blank, LCS, and either an US, D, MS or an LCS/LCSD.

C. QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data.

1. The limits are maintained in the LIMS for the relevant analysis numbers.
2. The limits are evaluated every 6 months and updated as needed.
3. The limits are subject to change without notification.

D. Batch Quality Control

1. For the preparation and concentrations of Batch Quality Control see Form 1−P−QM−FOR−9009076.
2. For the frequency, acceptance criteria and corrective action see Tables I, and II.

E. Raw data quality checks

1. Make sure that the run is correctly labeled, and dated and that the corresponding cover sheet is attached to the run.
2. Verify that the appropriate Tuning Report is with the run.
3. For run and batch QC/Calibration frequency, acceptance criteria and corrective action, see Method Specific Tables I (EPA 200.8), and II (EPA 6020). For information on statistical windows refer to 1−P−QM−QMA−9017313.
4. Spike levels of batch QC are available in the LIMS and on 1-P-QM-FOR-9009182.

5. LOQs are available to analysts in the LIMS.

6. Check to make sure that all results are not> 90% of the Linear Range (see Definition 16). If a sample reading is above 90% of the linear range, then reread the sample at a dilution sufficient to bring the sample concentration to approximately the middle of the calibration range.

7. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings <|−LOQ|.

8. Check for carryover between samples. Sample RSD >20%, with a concentration > the LOQ decreasing progressively over time (i.e., Reading 3<2<1). Flag any suspect samples for reread.

NOTE: Whenever a sample is encountered that exceeds the calibration range, the following sample must be checked for carryover for the over range analyte (s) and any suspect results must be deselected and reanalyzed. During reanalysis the sample order must be modified as needed to avoid carryover from any over range sample(s).

9. For all EW (samples from public drinking water sources), check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL notify a verifier at once. An automated email is sent to the Client Service Representative and the state for the analytes listed below with the exception of Pb and Cu which follow the 90th percentile rule (the CSR tracks the lead and copper and notifies the supplier when necessary). Suppliers must be notified within 24 hours.

10. ICP-MS is an analytical technique that can be subject to significant interference on certain analytes including but not limited to Cr, Ni, Cu, As, Se, and Cd. In prior versions of this procedure the approach used to deal with these types of interferences involved a significant amount of additional data review, including monitoring multiple masses, selecting alternate masses to avoid interference, comparison of suspect data to alternate techniques and additional measures. This approach is no longer necessary due to the effective removal of these types of interferences using collision cell ICP−MS.

11. To avoid the risks associated with polyatomic interferences in wastewater and soil matrices all analytes must be analyzed on the collision cell ICP−MS (Agilent 7500ce, Equipment #11332 OR Agilent 7700x, Equipment #19204) with the following exceptions:

    a. Be, Sb, Ba, Tl and Pb are also analyzed in wastewater and soil samples using the non-collision cell ICP−MS (Perkin Elmer ELAN 9000, Equipment #10007). These

analytes have historically been free of the types of interferences that are corrected using collision cell ICP−MS.

b. Because EPA 200.8 rev. 5.4 does not currently allow the analysis of drinking water samples using collision cell ICP-MS all drinking water samples must be analyzed using the non-collision cell ICP−MS (Perkin Elmer ELAN 9000, Equipment #10007) or by using a method with no gas mode for all analytes on either the Agilent 7500 or 7700. The following analytes are analyzed in drinking water samples: Be, Cr, Ni, Cu, As, Se, Ag, Cd, Sb, Tl, and Pb. Drinking water samples have also been historically free of the types of interferences that made alternate mass selection necessary; multiple masses are no longer used in the instrument method and only the masses referenced at the beginning of this procedure are used.

12. Check the internal standard level for the entire run. If the internal standard reading for any sample is out of the acceptance range of the S0 reading, evaluate whether this is a result of the sample matrix or drift from other samples in the run. Only reread the sample at a dilution if the internal standard appears to be out of range due to the sample matrix. For samples where an internal standard is above the acceptance range it is acceptable to reprocess the data using an alternate internal standard. This is the preferred approach in order to avoid raised reporting limits. The acceptance criteria differ based on the method being used. Refer to Table I, and II for the method acceptance criteria.

NOTE: The internal standard is added in equal concentration to all of the samples and standards via a dedicated line on the peristaltic pump. The analytical lines referenced to an internal standard report a corrected concentration value based on the ratio of analyte to internal standard intensities. All the calculations for determining concentration are based on Intensity Ratio (IR). The IR is defined as the background corrected intensity signal of the analyte line (Ia) divided by the internal standard value (Iis). IR= Ia/Iis.

13. For TCLP and SPLP samples, an MSA (method of standard additions) is required if:

a. The sample concentration falls between 80% and 100% of the regulatory limit.

b. If the TCLP or SPLP matrix spike (QA) recovers <20%, all samples in the leachate batch must be reanalyzed using the method of standard additions for that analyte.

14. When items 1 – 13 are complete, check the following:

a. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms.

b. The data are uploaded to LIMS via IDAT by the reviewer.

F. Verification process

1. Confirm that all required pieces of QC have been uploaded to LIMS and are within specification. If there is partial QC on the current run and the samples have been analyzed more than once, check to see if there are associated runs in the hold bin waiting for additional QC to be verified.

2. Choose method of verification. (Metals verification by run, verify by multiple elements per sample, or verify by individual element).

3. Check lab notes and project notes for each sample group.

4. If a technical decision or a client decision has been made to accept data that is not within specification, a nonconformance form Form1−P−QM−FOR−9007858 must be filled out and signed by the investigator and a verifier. An electronic copy of the nonconformance form must be placed in the nonconformance forms folder located under the dept 22 folder on lldata/env. Quality Assurance reviews these forms on a monthly basis. Notify your supervisor each time that a nonconformance form has been generated.

5. Once all of the elements are verified for a digest, verify the digest number. Suite and tracking numbers will auto verify after all the metals analyses are verified.

Figure I

Daily Performance Acceptance Criteria

In Sensitivity	>300,000 cps
Mg Sensitivity	>40,000 cps
Pb Sensitivity	>100,000 cps
CeO/Ce	≤0.03
Ba++/Ba+	≤0.03
Background	<30 cps @ Mass 220
Net RSd	≤%5.0

Table I

EPA 200.8

ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Tuning	Daily	No AMU diff. of >0.1 P.W. ≥0.64 and ≤0.66 (Elan 9000) P.W. < 0.9 at 10% height (Agilent 7500 and 7700) %RSD <5 for masses used for tuning	Perform mass calibration for AMU. Adjust mass calibration for P.W.
Daily Performance	Daily	Evaluated for information only	Instrument maintenance and optimization as needed.
Calibration	The calibration must contain a blank and 1 standard		
Calibration for samples that require or use a multipoint calibration	The calibration must contain a blank and three standards.	Correlation coefficient (r^2) for the curve must be ≥0.995.	Terminate run sequence and recalibrate. Data must not be reported for the analyte unless the correlation coefficient is ≥0.995
Initial Calibration Verification (ICV)	Must be analyzed immediately following calibration.	±10% of the true value	If the ICV is out of specification high and the result is not < - LOQ, accept results that report as non-detect for the affected analyte (s). Results for the affected analyte(s) ≥ to the reporting limit must not be reported (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be <3*IDL. If ICB is Out of Specification positive (+), accept results that are >10× the ICB, or < reporting limit. If ICB is Out of Specification negative (-), only accept results that are >10× ICB.	Data for that analyte must not be reported from the run (reanalyze).
Low Level Check (LLC)	Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB.	+/- 50% of the true value. Not applicable if sample concentrations are >10× the true value of the LLC.	Data for that analyte must not be reported from the sample (reanalyze).
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA must be analyzed at the beginning and end of each run immediately following the LLC. The ICSAB must be analyzed at the beginning and end of each run immediately following the ICSA.	±20% of the true value for the analytes that are spiked. |ICSA| or |ICSAB| must be <2× LOQ for analytes that are not spiked.	Data for that analyte must not be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples	±15% of the true value.	If the CCV is out of specification and the result is not < - LOQ, accept results that report as non-detect for the affected analyte(s). Results for the affected analyte(s) ≥ to the reporting

	Frequency	Acceptance	Corrective Action
			limit must not be reported (reanalyze).

Table I – Continued
EPA 200.8

ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples	|CCB| must be <3*IDL. If CCB is Out of Specification positive (+), accept results that are >10× the CCB, or < reporting limit. If CCB is Out of Specification negative (-), only accept results that are >10× CCB.	Data bracketing the CCB for the affected analyte must not be reported (reanalyze).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	|PB| must be < ½ LOQ or 2.2× MDL whichever is greater. Does not apply to samples >10× blank value. EW samples are not accepted for any reason if the PB is out of specification.	Redigest all associated samples. EW samples must be redigested.
Laboratory Fortified Blank (LCS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less. *Note: The LCS is spiked at or below the MCL for all primary drinking water metals. See QA/QC section E.9. for MCL levels.	Use statistical limits or the method limit of ±15%,, as indicated by the client requirement. EW samples are not accepted if the LCS is out of specification.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data is acceptable. EW samples must be redigested.
Laboratory Fortified Blank Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less. *Note: The LCS is spiked at or below the MCL for all primary drinking water metals. See QA/QC section E.9. for MCL levels.	Use statistical limits or the method limit of ±15%, as indicated by the client requirement. EW samples are not accepted if the LCS is out of specification. RPD must be <20%.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data is acceptable. EW samples must be redigested. Redigest if RPD is out of specification.
Matrix Spike (MS)	Rate of 10% of analytical samples	Use statistical limits or the method limit of ±30%, as	

	Frequency	Acceptance	Corrective Action
		indicated by the client requirement.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less	If the samples are >5× the LOQ the RPD must be <20%	'*'-Flag the data package forms.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	±15%	Report % rec. on FormVB-IN.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50 MDL.	The percent difference must be <10%	'E'- Flag data on DP Forms

Table I – Continued
EPA 200.8

ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Samples		Sample reading must be within the linear range (see Definition 16). Elements reported as non-detect are accepted if the ICV/CCV is out of specification high, and the sample is not < - LOQ. RSD must be <20% for results >2× LOQ.	Reanalyze at dilution that brings sample concentration within 90% of the linear range. Reanalyze for elements that do not meet this criteria. Reanalyze for elements that do not meet this criteria.
Linear Range (LR)	Analyzed quarterly.	±10% of the true value	Samples reading greater than 90% of the calibration range must be reanalyzed.
Internal Standards	Added to all samples by way of second pump channel.	Must be 60%-125% of the Calibration Blank	Reanalyze @ DF2

Table II

EPA 6020/6020A/6020B

ICP-MS Metals

	Frequency	Acceptance	Corrective Action
uning	Daily	No AMU diff. of >0.1 P.W. ≥0.64 and ≤0.66 (Elan 9000 only) P.W. < 0.9 at 10% height (Agilent 7500 and 7700) %RSD <5 for masses used for tuning	Perform mass calibration for AMU. Adjust mass calibration for P.W.
aily Performance	Daily	Evaluated for information only	Instrument maintenance and optimization as needed.
alibration	The calibration must contain a blank and 1 standard.		
alibration for samples at require or use a ultipoint calibration	The calibration must contain a blank and three standards.	For EPA 6020/6020B: Correlation coefficient (r^2) for the curve must be ≥0.995. For EPA 6020A: Correlation coefficient (r^2) for the curve must be ≥0.998.	For EPA 6020: Terminate run sequence and recalibrate. Data must not be reported for the analyte unless the correlation coefficient is ≥0.995 For EPA 6020A/6020B: Terminate run sequence and recalibrate. Data must not be reported for the analyte unless the correlation coefficient is ≥0.998
itial Calibration erification (ICV)	Must be analyzed immediately following calibration.	±10% of the true value	If the ICV is out of specification high and the result is not < -LOQ, accept results that report as non-detect for the affected analyte(s). Results for the affected analyte(s) ≥ to the reporting limit must not be reported (reanalyze).
itial Calibration Blank CB)	Must be analyzed immediately following the ICV.	For 6020/6020A: |ICB| must be <3*IDL. For 6020B: |ICB| must be < ½ LOQ If ICB is Out of Specification positive (+), accept results that are >10× the ICB, or < reporting limit. If ICB is Out of Specification negative (-), only accept results that are >10× ICB.	Data for that analyte must not be reported from the run (reanalyze).

	Frequency	Acceptance	Corrective Action
ow Level Check (LLC)	6020/6020A: Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB 6020B: Must be analyzed at the beginning of each run.	For 6020:±50% of the true value Not applicable if sample concentrations are >10× the true value of the LLC For 6020A: ±30% of the true value Not applicable if sample concentrations are < LOQ, or if sample concentrations are greater than the CCV (CCV must be within specification). For 6020B: ±20% of the true value Not applicable if sample concentrations are < LOQ, or if sample concentrations are greater than the CCV (CCV must be within specification).	Data for that analyte must not be reported from the sample (reanalyze).

Table II - Continued
EPA 6020/6020A/6020B

ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Interference Check Standard A and AB (ICSA/ICSAB)	6020/6020A: The ICSA must be analyzed at the beginning and end of each run immediately following the LLC. The ICSAB must be analyzed at the beginning and end of each run immediately following the ICSA. 6020B: ICSA only required (run on every run only at the beginning of each run immediately following the LLC.	6020/6020A: ±20% of the true value for analytes that are spiked. |ICSA| or |ICSAB| must be <2× LOQ for analytes that are not spiked. 6020B: |ICSA| must be < LOQ for analytes that are not spiked.	Data for that analyte must not be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples	±10% of the true value.	If the CCV is out of specification high and the result is not < - LOQ, accept results that report as non-detect for the affected analyte

			(s). Results for the affected analyte(s) ≥ to the reporting limit must not be reported (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples	6020/6020A: |CCB| must be <3*IDL. 6020B: |CCB| must be < ½ LOQ If CCB is Out of Specification positive (+), accept results that are >10× the CCB, or < reporting limit. If CCB is Out of Specification negative (-), only accept results that are >10× CCB.	Data bracketing the CCB for the affected analyte must not be reported (reanalyze).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	|PB| must be < ½ LOQ For 6020: Not applicable if analyte reading in the sample is >20× the PB reading or < the reporting limit. For 6020A/6020B: Not applicable if analyte reading in the sample is >10× the PB reading or < the reporting limit.	Redigest all associated samples.
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data is acceptable.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement. RPD must be <20%.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data is acceptable. Redigest if RPD is out of specification.

Table II - Continued
EPA 6020/6020A/6020B

ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Must be prepped at a frequency of 1 per	Use statistical limits or the method limit of ±25%, as	

	analytical batch of 20 samples or less.	indicated by the client requirement. RPD must be <20%	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less	If the samples are >5× the LOQ the RPD must be <20%.	Flagged in data package and in the QC Summary.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	±15% of the true value	The data is flagged in the data package.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >100× MDL.	The percent difference must be <10%.	The data is flagged in the data package.
Samples		Sample reading must be within the linear range (see Definition 16). Elements reported as non-detect are accepted if the ICV/CCV is out of specification high, and the sample is not < - LOQ. RSD must be <20% for results >2× LOQ.	Reanalyze at dilution that brings sample concentration within 90% of the linear range. Reanalyze for elements that do not meet this criteria. Reanalyze for elements that do not meet this criteria.
Linear Range (LR)	Analyzed quarterly	±10% of the true value	Samples reading greater than 90% of the calibration range must be reanalyzed.
Upper Linear Range	6020B only. Analyzed once per run	±10% of the true value	Samples reading greater than 90% of the calibration range must be reanalyzed.
Mid Range Check (1/2 the Upper Linear Range)	6020B only. Analyzed once per run.	±10% of the true value	Reanalyze all elements > |CCV|.
Internal Standards	Added to everything through use of second pump channel	For 6020: 30%-120% for samples 80%-120% for ICV, CCVs, ICB, and CCBs. 30%-120% for LLC, ICSA, and ICSAB. For 6020A/6020B: 70-130% for samples.	Reanalyze @ DF5. Terminate analysis and reanalyze. Reanalyze. Reanalyze @ DF5.

End of document

Version history

Version	Approval	Revision information
6	15.JAN.2015	
7	29.JUN.2017	

eurofins	Always check on-line for validity **Sample Preparation of Leachates and Other Wastewater for Analysis of Total Metals by Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)**	Level: **Work Instruction**
Document number: **T-MET-WI11937**		
Old Reference: **1-P-QM-WI-9015165**		
Version: **12**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **19-JAN-2015**	Document users: **6_EUUSLA_Metals_ICP-MS Prep, 6_EUUSLA_Metals_ICP-MS Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis DOD - 6050, 10639

Revision Log

Revision: 12		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Reference	No longer applicable to the procedure	Removed 3010A Modified
Reference Modifications	No longer applicable to the procedure	Removed 3010A modifications
Procedure A and B	Clarification	Reworded numbers 2, 5 and 6 to clarify steps found in the EPA procedure for 3020A

Revision: 11	**Effective Date:** Nov 25, 2013	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Cross Reference	Reflect current procedure	Added reference to Analysis #6142, 6123, 6125,…
Sample Collection, Preservation, and Handling	Process change	Changed sample storage temperature from 4° ± 2° C to 0° to 6° C but not frozen, prior to digestion.
Safety Precautions and Waste Handling	No longer used	Deleted text pertaining to Hydrofluoric Acid.
Reagent and Standards	Reflect current procedure	Added text pertaining to preparing solutions using different volumes, is acceptable, if exact ratios are maintained.
Procedure A	Reflect current procedure	Deleted text, in NOTE, pertaining to using a smaller sample aliquot if insufficient sample is submitted. Deleted text, to NOTE, pertaining to samples concentration
Procedure A.1	Reflect current procedure	Added text pertaining to adding spike solution, after the sample has been poured. Added text pertaining to reference to Analysis #6142, 6123, 6125,… for batch quality control requirements.
Procedure B	Reflect current procedure	Deleted text in NOTE pertaining to using a smaller sample aliquot if insufficient sample is submitted. Deleted text to NOTE pertaining to samples concentration
Procedure B.1	Reflect current procedure	Added text pertaining to reference to Analysis #6142, 6123, 6125,… for batch quality control
Block Digestor Instructions	Reflect current procedure	Clarified instruction steps. Deleted text pertaining to the difference between sample temperature and display temperature.
Quality Assurance/Quality Control	Reflect current procedure	Added reference to Analysis #6142, 6123, 6125,… for batch quality control requirements.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3020A, July 1992.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A (aqueous, solid, tissue) and EPA 200.8 (aqueous)

Document	Document Title
1-P-QM-FOR-9009182	Working Instructions for Prep Solutions and Standards
1-P-QM-QMA-9015390	Demonstrations of Capability

Purpose

This digestion procedure is used to prepare leachate and other wastewater samples for measurement of total metals by inductively coupled plasma-mass spectrometer (ICP-MS) following SW 846 protocol.

Scope

This acid digestion procedure is used by the Metals Department of the Environmental Sciences Division to prepare leachate, wastewater, surface water, and groundwater samples for measurement of total recoverable metals by inductively coupled plasma-Mass Spectrometer (ICP-MS) following SW-846.

Basic Principles

A mixture of nitric acid and the sample is refluxed in a covered beaker/digestion vessel at low volume to dissolve metals. It is cooled and brought up to volume with reagent water.

Reference Modifications

1. A 50-mL sample aliquot and final volume is used instead of 100-mL to improve digestion throughput, conserve sample usage, and limit waste generation. Because all reagents are also adjusted so that concentrations are equivalent to a 100-mL aliquot, there is no impact on the data.

2. Ribbed watch glasses are not used; samples are evaporated without watch glasses in nonmetallic hoods to speed evaporation. No contamination trends have been observed in prep blanks evaporated without watch glasses.

Definitions

1. ACS – American Chemical Society
2. ASTM – American Society of Testing and Materials
3. D – Sample Duplicate
4. DOC – Demonstration of Capability
5. IDOC – Initial Demonstration of Capability

6. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate

7. LCSW – Laboratory Control Sample Water

8. LLENS – the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the LIMS.

9. LIMS – Laboratory Information Management Systems

10. LLI Sample ID – unique 7-digit number assigned to a client sample.

11. LOQ – Limit of Quantitation

12. MDL – Method Detection Limit

13. MS (R) – Matrix Spike

14. MSD (M) – Matrix spike duplicate

15. PB/PBW – Preparation Blank/ Preparation Blank Water

16. QC – Quality Control

17. Method Blank – equivalent to a Preparation Blank. A designated sample designed to monitor for sample contamination during the analysis process. A volume of reagent laboratory water is typically used to monitor water sample analysis, while solids blanks consist of a purified solid matrix or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

18. SOP – Standard Operating Procedure

19. SPLP – Synthetic Precipitation Leaching Procedure

20. STLC – Soluble Threshold Limit Concentration

21. TCLP – Toxicity Characteristic Leaching Procedure

22. U or US – unspiked background sample

Interferences

Not applicable to this procedure.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

> **Nitric acid (HNO_3)** – This acid can cause skin burns. Add nitric acid to samples in a hood or use the designated dispensing equipment to avoid exposure to toxic fumes.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A , soda ash or equivalent, to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf. Soda ash is located in the stairwell adjacent to the prep room.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability for this or an equivalent procedure.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documentation of the critical steps in this procedure, over checking of data as well as an IDOC.

The IDOC and the DOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Refer to 1-P-QM-QMA-9015390, for specific requirements. A DOC is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

Samples are collected in plastic containers and preserved to a pH of <2 with HNO_3. (Samples to be analyzed for soluble metals requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45-micron filter within 5 days of receipt and then preserved.) The pH is checked upon receipt and adjusted as necessary by Sample Support; samples that are pH adjusted at the lab must not be digested for a minimum of 24 hours. If samples fail to maintain a pH of <2 the Client Service Representative is notified for further direction. Samples are stored at 0° - 6°C, but not frozen, prior to digestion. Samples must be digested within 6 months of collection. Digested samples are stored in plastic at room temperature and have a 6 month holding time.

Apparatus and Equipment

1. Polypropylene containers (digestion vessels) – certified clean and Class A equivalent
2. Watch glasses or reflux caps
3. 50-mL graduated cylinders or other appropriate graduated cylinders if necessary
4. 50-mL volumetric flasks or other appropriate Class A volumetric flasks if necessary
5. 250-mL beakers or other appropriate beakers
6. Hotblocks or hot plates, adjustable and capable of maintaining a temperature of 90° to 95°C

Reagents and Standards

For reagent preparation, shelf life, and storage conditions, see Form 1-P-QM-FOR-9009182. .

Nitric acid, HNO_3 – Fisher, Trace Metal Grade, or equivalent. Store at room temperature and re-evaluate annually.

NOTE: It is acceptable to prepare solutions using multiples of indicated volumes if exact ratios are maintained.

Calibration

Not applicable to this method.

Procedure

This SOP has been set up to outline the procedures for both hotblock and hot plate digestions (see below). Choose the procedure that corresponds to the sample heating technique being used for sample digestion.

A. Hotblock

NOTE: When insoluble matter is present in the digested sample, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45−micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered water.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

1. Shake sample well. Transfer 50 mL of well mixed sample to a 68−mL digestion vessel. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1−P−QM−FOR−9009182. For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

NOTE: For leachate samples, use the appropriate extraction fluid for the PBW and LCS. The extraction fluids are as follows: TCLP, SPLP, STLC, ASTM, Filtration, and Elutriate.

2. Add 1.5 mL of HNO_3. Place the vessel in the hotblock at 90° to 95°C, and cautiously evaporate to low volume (about 5 mL), making certain that the sample does not boil and that no portion of the bottom of the digestion vessel is allowed to go dry.

3. Cool the digestion vessel and add another 1.5–mL portion of HNO_3.

4. Cover the digestion vessel with a reflux cap and return to the hotblock. Increase the temperature of the hotblock so that gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries result. If this occurs, discard the sample and re-prepare in a new batch.

5. Continue heating (refluxing), adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

6. When digestion is complete, uncover the digestion vessel and evaporate to low volume (about 3 mL). Do not allow any portion of the bottom of the digestion vessel to go dry.

7. Remove the digestion vessel and add approximately 5 mL of reagent water, mix, and continue warming for 10 to 15 minutes to allow additional solubilization of any residue to occur.

8. Allow to cool. Adjust volume to the 50–mL mark on the digestion vessel with reagent water and mix. Seal vessel with screw cap.

9. The sample is now ready for analysis.

B. Hot Plates

NOTE: When insoluble matter is present in the digested sample, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45–micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered reagent water.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

1. Shake sample well. Use a 50 mL graduated cylinder to transfer 50 mL of well mixed sample into a 250 mL beaker. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1–P–QM–FOR–9009182. For sample batch quality control requirements

see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

NOTE: For leachate samples, use the appropriate extraction fluid for the PBW and LCS. The extraction fluid are as follows: TCLP, SPLP, STLC, ASTM, Filtration, and Elutriate.

2. Add 1.5 mL of HNO_3. Place the beaker on a hot plate and cautiously evaporate to low volume (about 5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry.

3. Cool the beaker and add another 1.5–mL portion of HNO_3.

4. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries result. If this occurs, discard the sample and reprepare in a new batch.

5. Continue heating (refluxing), adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

6. When digestion is complete, uncover the beaker and evaporate to low volume (about 3 mL). Do not allow any portion of the bottom of the beaker to go dry.

7. Remove the beaker and add approximately 5 mL of reagent water, mix, and continue warming the beaker for 10 to 15 minutes to allow additional solubilization of any residue to occur.

8. Allow to cool. Transfer the solution to a 50–mL volumetric flask. Adjust volume to the 50 mL mark with reagent water and mix.

9. Transfer to a polypropylene bottle.

10. The sample is now ready for analysis.

Block Digestion Instructions

1. Turn block digestor on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. PRESS and hold STAR (*) key.

4. The display shows the Set Point Temperature.

5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 90° to 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations
Not applicable to this procedure.

Statistical Information/Method Performance
Not applicable to this method. See analysis procedure.

Quality Assurance/Quality Control
A method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample must be performed with every digestion batch (20 samples or less). Each piece of batch QC is digested following the procedure in this SOP.

For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

End of document

Version history

Version	Approval	Revision information
12	19.JAN.2015	

eurofins	Always check on-line for validity **Preparation of Solids by EPA 7471A or B for Mercury Analysis**	Level: **Work Instruction**
Document number: **T-MET-WI11948**		
Old Reference: **1-P-QM-WI-9015161**		
Version: **19**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **19-JUL-2016**	Document users: **6_EUUSLA_Metals_Hg Prep Verifiers, 6_EUUSLA_Metals_Hr Prep, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis DOD - 5711, 10638

Revision Log

Revision: 19		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Enhancement	Add method. Change title to: Preparation of Solids by EPA 7471A or B for Mercury Analysis
Reference	No change in chemistry	Removed the word Modified.
Purpose	Optional section. Information is contained within the scope	Removed section.
Definitions	Optional section which is unnecessary	Removed section.
Reagents and Standards	Enhancement	Added 40ppb Hg water standard and 100ppb Hg soil standard.

eurofins	Always check on-line for validity **Preparation of Solids by EPA 7471A or B for Mercury Analysis**	Level: **Work Instruction**
Document number: **T-MET-WI11948**		
Old Reference: **1-P-QM-WI-9015161**		
Version: **19**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **19-JUL-2016**	Document users: **6_EUUSLA_Metals_Hg Prep Verifiers, 6_EUUSLA_Metals_Hr Prep, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

Revision: 19	**Effective Date:** This version	
Procedure	Reflects current procedure	Added digestion of wipes.
	Clarification	Added text pertaining to bringing temp to 95ºC and then time the 2 minutes.
	Clarification	Added text pertaining to carefully swirl container after addition of KMNO4.

Revision: 18	**Effective Date:** Aug 28, 2014	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Clarification	Removed Fish from the title so that the word Tissue is not limited to only fish.
Sample Collection, Preservation, and Handling	No longer applicable. Laboratory is not supporting CLP work.	Deleted holding time of 26 days for CLP
Procedure 2	Clarification	Removed Fish from the tissue section and included or other tissue samples are used.
Procedure 6	Clarification	Added the word about. To read: …adding about 5 ml of aqua regia
Purpose	Clarification	Removed the word fish so that the word tissue is not limited to only fish.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471A, Rev. 1, September 1994.
2. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471B, Rev. 2, February 2007
3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0259, 0159	Mercury in Aqueous, Solid and Tissue Samples by Cold Vapor AA
1-P-QM-FOR-9008921	Working Instructions for Preparation of Mercury Solutions and Standards

Scope

This procedure is used to prepare soil, sediment, sludge, oil, tissue and wipe samples for the measurement of mercury by atomic absorption cold vapor technique following SW-846 7471A or 7471B protocol. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix. Samples that require additional homogenization are addressed on a case-by-case basis and homogenized by the Sample Support Group (Department 6055).

Basic Principles

Samples are digested with aqua regia and potassium permanganate to oxidize mercury compounds to mercuric ions and eliminate possible interference from sulfide. Samples high in chlorides require additional permanganate. At the time of analysis, excess permanganate is reduced with sodium chloride/hydroxylamine hydrochloride. Mercuric ions are reduced to mercury metal using stannous chloride. Mercury measurement is performed using mercury cold vapor technique.

Reference Modifications

To increase efficiency, polypropylene containers are used in place of BOD bottles. Prior to analysis (after excess potassium permanganate is reduced with sodium chloride/hydroxylamine hydrochloride solution) samples are adjusted to 100 mL in volumetric flasks. This allows aliquots to be taken as required for analysis; aliquots cannot be taken when BOD bottles are used. No impact on the quality of the data generated using this modification has been observed.

Interferences

Not applicable to this procedure.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO_3) – This acid can cause skin burns. Add nitric acid to samples in a hood or use the designated dispensing equipment to avoid exposure to toxic fumes.

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H_2SO_4 to avoid a violent reaction. Always use in a fume hood or use the designated dispensing equipment.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure.

Proficiency is measured through an Initial Demonstration of Capability (IDOC) that consists of four laboratory control samples (LCS) that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four LCS's or one blind sample.

Sample Collection, Preservation, and Handling

Samples are collected in either glass or plastic containers with no preservatives. They must be stored at 0° to 6°C, not frozen and digested and analyzed within 28 days of collection.

Digested samples are stored in plastic containers at room temperature. Store samples, standards, and digested samples separately.

Apparatus and Equipment

1. Polypropylene containers (digestion vessels) - Certified clean and Class A equivalent

2. Balance, capable of reading 0.1 mg

3. Polypropylene covers, (digestion vessel covers)

4. Chemware Ultra-Pure PTFE boiling stones, or equivalent

5. Environmental Express Hotblock (block digestor), adjustable and capable of maintaining a temperature of 95º ± 1°C

6. LLENS (Lancaster Laboratories Electronic Notebook System) - the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the laboratory information management system (LIMS).

Reagents and Standards

A. Store all prepared standards and reagents in glass or polyethylene bottles at room temperature. Label the bottle with the solution name, lot number, date prepared, the expiration date, the initials of the person preparing the solution, and the storage conditions.

B. Standard/ spiking concentration and reagent vendors are subject to change without notification.

C. Reagents - Follow manufacturer's storage conditions and expiration date. If no expiration date is provided, re-evaluate annually or set a one year expiration date. Use the following or equivalent:

1. Hydrochloric acid, 36.5% to 38.0% HCl, Fisher Trace Metal Grade reagent, 1.194 g/mL

2. Nitric acid, 70.0% to 71.0% HNO_3, Fisher Trace Metal Grade reagent, 1.428 g/mL

3. Potassium permanganate, $KMNO_4$, Baker Analyzed reagent, ACS.

4. Sodium chloride, NaCl, J.T. Baker, Certified ACS.

5. Hydroxylamine hydrochloride, $NH_2OH{\bullet}HCl$, J.T. Baker, Certified ACS.

6. 1000 mg/L Hg standard solution, Baker analyzed reagent.

D. Working instructions for the preparation of mercury solutions and standards are contained within Form 1−P−QM−FOR−9008921. This form includes shelf life and storage conditions.

1. Hg intermediate standard (10 mg/L).
2. Hg intermediate standard (1.0 mg/L).
3. 40 ppb Hg Water Standard.
4. 100 ppb Hg Soil Standard.
5. Potassium permanganate solution (5%).
6. Aqua regia.
7. Sodium chloride/hydroxylamine hydrochloride solution.

E. Adjust all additions according to final solution volume if larger or smaller volumes are needed. Thoroughly mix the solution after diluting to volume.

Calibration
Not applicable to this procedure.

Block Digestor Instructions
1. Turn block digestor on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. PRESS and hold STAR (*) key.

4. The display shows the Set Point Temperature.

5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 95° ± 1°C sample temperature.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting.

Procedure

1. Turn block digestor on and allow block to reach the Control Point setting that provides 95° ± 1°C sample temperature.

2. Weigh three aliquots, ~ 0.2000g each, taken from three different areas (combined 0.6000 to 0.6500 g to the nearest 0.0001 g) of a well mixed, as-received sample into a polypropylene digestion vessel.

   a. Add 0.6000g to 0.6499g of Chemware Ultra-Pure PTFE boiling stones to the vessel for the method blank and LCS.

   b. Enter the blank weight as 0.6000 with final volume of 100.0000.and the LCS weight as 1.0000 with final volume 100.0000 in the LLENS.

3. Matrix exceptions:

   a. Samples with a liquid consistency: increase weight to 1 g (1.00 to 1.05 g).

   b. Oil samples: weigh 0.2000 g to 0.2500 g (to the nearest 0.0001 g) of sample and add 0.2000g to 0.2499g of Teflon Chips to the blank and LCS container. Enter the blank weight as 0.2000 to 100.0000 final volume and the LCS weight as 1.0000 to 100.0000 final volume in the LLENS.

   c. Wipes: one blank media each must be used for the batch preparation blank, the LCS, and the laboratory control sample duplicate (LCSD). Use reagent water to rinse any particulate matter from the wipe container into the vessel containing the wipe before digesting. Digest wipes in their own batch.

   d. Tissue samples: spike the LCS, LCSD in the same manner as the matrix spike and matrix spike duplicate (Refer to Form 1-P-QM-FOR-9008921). Digest tissue samples in their own batch.

4. For sample batch quality control preparation, spiking procedures and concentration levels see Form 1-P-QM-FOR-9008921.

5. All spiking must be performed prior to starting the digestion procedure.

6. Add about 5 mL reagent water and about 5 mL of aqua regia solution.

7. Place sample containers in block digestor, bring to 95ºC ± 1°C and heat approximately 2 minutes. (Place a calibrated thermometer in batch blank container.)

8. Remove sample containers from block and allow to cool.

9. Add 50 mL of reagent water and 15 mL of 5% $KMnO_4$ solution and mix by carefully swirling the container.

a. Add additional portions of 5% $KMnO_4$ solution (in 5−mL increments, up to as much as 25mL), if necessary, until the purple color persists for at least 15 minutes.

b. Add the same amount of $KMnO_4$ solution to entire digestion batch.

c If the maximum amount of 25mL of 5% KMnO4 solution was added and the purple color did not persist for at least 15 minutes, then contact group leader, further dilutions are required. A comment must be placed on the batch sheet documenting the reason for the dilution.

10. Transfer sample containers to block digestor.

11. Place a calibrated thermometer in batch blank container.

12. Put a polypropylene cover on each container.

13. When the thermometer indicates 95° ± 1°C, continue heating for 30 minutes.

14. Remove sample containers from digestion block and allow to cool. Seal container with screw cap.

15. The sample is now ready for analysis.

Calculations
Not applicable to this procedure.

Statistical Information/Method Performance
Not applicable to this method. See analysis method.

Quality Assurance/Quality Control
Each digestion batch (up to 20 samples) must contain a method blank (PB), LCS and either an unspiked background sample (US), duplicate (D), matrix spike (R), matrix spike duplicate (M) or an LCS/LCSD.

Refer to Analysis #0259, 0159 for sample batch quality control requirements, acceptance criteria and corrective action.

End of document

eurofins	Always check on-line for validity **Preparation of Solids by EPA 7471A or B for Mercury Analysis**	Level: **Work Instruction**
Document number: **T-MET-WI11948**		
Old Reference: **1-P-QM-WI-9015161**		
Version: **19**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **19-JUL-2016**	Document users: **6_EUUSLA_Metals_Hg Prep Verifiers, 6_EUUSLA_Metals_Hr Prep, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

Version history

Version	Approval	Revision information
19	19.JUL.2016	

eurofins	Always check on-line for validity **Mercury in Aqueous, Solid and Tissue Samples by Cold Vapor AA**	Level: **Work Instruction**
Document number: **T-MET-WI7965**		
Old Reference: **1-P-QM-WI-9015067**		
Version: **16**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **27-NOV-2015**	Document users: **6_EUUSLA_Metals_Hg Analysis, 6_EUUSLA_Metals_Hg Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis DOD - 0259, 0159

Table of Contents

Table of Contents

Revision Log

Revision: 16	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Personnel Training and Qualifications	Enhancement	Added information on IDOC and DOC options.
Table 1	Clarification for compliance	Added a note that the LCS is spiked at or below the MCL for drinking water.
Table 1	Clarification for compliance	Added new EW rule for the PB and LCS if they are out of specification data cannot be accepted for any reason.
Table 1 and 2	Clarification for compliance	PB requirements acceptance criteria updated.

Revision: 15	**Effective Date:** Jun 16, 2015	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	No longer applicable Clarification	Removed CLP references. Added eLIMS-EP for Parallax.
Table I and II	Clarification	Clarified the acceptance criteria for LCS/LCSD.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 7470A, September 1994

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471B, February 2007.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471A, September 1994

4 Method 245.1 (rev. 3), Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectroscopy, USEPA 600/R-94/111 May 1994.

5. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #5711, 10638	Sample Preparation of Soil, Sediment, Sludge, Oils, and Tissues for Total Mercury Analysis by Atomic Absorption Cold Vapor Technique
Analysis #5713, 5714	Digestion of Aqueous Samples by SW-846 Method 7470A, EPA 254.1.

Document	Document Title
1-P-QM-FOR-9007858	Nonconformance Form
1-P-QM-FOR-9008921	Working Instructions for Preparation of Mercury Solutions and Standards
1-P-QM-QMA-9015390	Demonstrations of Capability
1-P-QM-QMA-9017325	Instrument and Equipment Maintenance and Calibration

Purpose
The purpose of this SOP is to describe the proper analysis of aqueous, solid and tissue samples for Mercury by Cold Vapor Atomic Absorption.

Scope
This method is used for determination of mercury in aqueous and solid samples. The optimum concentration range for this method is 0.2 to 5.0 ppb.

Matrices - EPA 7470A is applicable to water analysis. EPA 7471A and EPA 7471B are applicable to soil and tissue analysis. EPA 245.1 is applicable to water analysis.

LOQs are based on annual statistical evaluation of laboratory data and are subject to change. The current MDLs and LOQs are maintained in the LIMS.

Limits of Quantitation are subject to change without notification.

Background Information
Not applicable

Basic Principles
The Leeman Labs Mercury Analyzer utilizes continuous flow technology with drying of the sample vapor for the analysis of mercury by automated vapor generation. The reaction for the mercury analysis is a simple reduction reaction. The mercury is reduced with stannous chloride to liberate mercury metal and Tin (IV) chloride. An inert gas is used to sweep the volatile mercury into the absorption cell in the optical path of the atomic absorption spectrophotometer. The dry vapor enters one path of the optical cell, which has been optimized for fast response (small diameter), and sensitivity (long length).

Mercury is measured using a solid state detector with a wide dynamic range and a mercury source that delivers a stable source of emission at 254 nm. The signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions.

Reference Modifications

SW-846 Methods 7470A, 7471A, 7471B and EPA 245.1 are manual procedures. This SOP is written for an automated determination. The chemistry used to perform the mercury determination is the same. This modification does not impact the quality of the data generated.

Definitions

1. 0.15% HNO_3 – 0.15% Nitric Acid Solution
2. ACS – American Chemical Society
3. Calibration Blanks – includes ICBs and CCBs
4. CCB – Continuing Calibration Blank
5. CCV – Continuing Calibration Verification
6. CRA – Low Level Check Standard
7. D – Sample Duplicate
8. DOC – Demonstration of Capability
9. ICB – Initial Calibration Blank
10. ICV – Initial Calibration Verification
11. IDOC – Initial Demonstration of Capability
12. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate
13. LCSW/LCSS – Laboratory Control Sample Water/Laboratory Control Sample Solid
14. LIMS – Laboratory Information Management Systems
15. LLI Sample ID – unique 7-digit number assigned to a client sample.

16. LOQ – Limit of Quantitation

17. M – Sample spike duplicate

18. MDL – Method Detection Limit

19. MS/MSD – Matrix spike/matrix spike duplicate

20. PB/PBW/ PBS –Preparation Blank/ Preparation Blank Water/Preparation Blank Solid.

21. QC – Quality Control

22. R – sample spike

23. RPD – Relative Percent Difference

24. Leeman Labs Envoy software – a windows based program to help navigate the software.

25. Method Blank - equivalent to a Preparation Blank. A designated sample designed to monitor for sample contamination during the analysis process. A volume of reagent laboratory water is typically used to monitor water sample analysis, while solids blanks consist of a purified solid matrix or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

26. MSA – Method of Standard Additions

27. eLIMS-EP (Parallax) – The computer system that is used for Environmental work to track client samples and report results for those samples, unless spreadsheets or certificates of analysis reports are attached by the technical department. Also referred to as the LIMS.

28. SOP- Standard Operating Procedure

29. SPLP - Synthetic Precipitation Leaching Procedure

30. TCLP – Toxicity Characteristic Leaching Procedure

31. U or US – unspiked background sample

Interferences

Potassium permanganate is added to samples to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Take care to ensure that free chlorine is absent before the mercury is reduced and swept into the cell by using an excess of hydroxylamine sulfate (or chloride) reagent.

Copper has been reported to interfere; however, copper concentrations as high as 10 mg/kg had no effect on recovery of mercury from spiked samples.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

Sulfuric acid (H2SO4) – This acid is a strong oxidizing agent and can cause severe burns. Sulfuric acid spills are extremely slippery, adding to the danger. Always use in a fume hood. Never mix with concentrated HCl or concentrated KMNO4 to avoid a violent reaction (explosive splattering and extreme heat).

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds. Samples that contain dust may be hazardous. Open in a fume hood.

Samples that may contain cyanide require special precautions to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the system to set up sequences, perform the calculations, interpret raw data, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample. Refer to 1-P-QM-QMA-9015390 (DOC) for more guidance on these options.

Sample Collection, Preservation, and Handling

Aqueous samples are collected in plastic or glass containers, preserved to a pH of <2 with nitric acid and stored at 0° to 6°C not frozen. Samples must be digested within 28 days of collection for SW-846 Methods 7470A, 7471A, 7471B.

Drinking Water samples are collected in 1-L plastic or glass containers, preserved to a pH of <2 with nitric acid and stored at 0° to 6°C not frozen. Samples must be digested and analyzed within 28 days of collection for EPA 245.1

Solid samples are collected in glass containers and stored at 0° to 6°C not frozen. Samples must be digested and analyzed within 28 days of collection.

Dissolved Mercury: Samples to be analyzed for soluble mercury requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt and then for aqueous samples, samples are collected in plastic containers and preserved to a pH of <2 with HNO_3.

Store sample digestates in plastic bottles at room temperature. Store standards and digestates separately.

Apparatus and Equipment

Hydra II Mercury Analyzer with Envoy instrument software.

Reagents and Standards

A. Store all standards and reagents in polyethylene or glass containers at room temperature. Label the container with the solution name, lot number, date prepared, the expiration date, the initials of the person preparing the solution, and the storage conditions.

NOTE: Standard/ spiking concentration and reagent vendors are subject to change without notification.

B. Reagents use the following or equivalent:

1. Nitric acid, 70.0% to 71.0% HNO_3, Fisher Trace Metal Grade reagent, 1.428 g/mL; Store in glass container at room temperature. Follow manufacturer's expiration date.

2. Sodium chloride, NaCl, J.T. Baker, Certified ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.

3. Hydroxylamine hydrochloride, $NH_2OH{\bullet}HCl$, J.T. Baker, Certified ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.

4. Reagent Water

5. Stannous chloride solution, 10% SnCl, Baker Analyzed reagent, ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.

6. Hydrochloric acid, HCl, 36.5% to 38.0%, Fisher Trace Metal Grade reagent, 1.194 g/mL or equivalent. Store in glass container at room temperature. Follow manufacturer's expiration date.

C. For the preparation of calibration blanks, ICBs, CCBs, calibration standards, ICVs, CCVs, CRAs, Method Blanks, LCSs and Matrix Spikes solutions, see Form 1−P−QM−FOR−9008921.

D. General solutions - See Form 1−P−QM−FOR−9008921.

Calibration

A. Leeman Labs Hydra II Mercury Analyzer

1. The software program has been developed to check the correlation coefficient of the curve, run appropriate ICV and CCVs at proper intervals, and check the percent recoveries of the ICV and CCVs.

2. A recalibration and reread of any associated samples is required for any checks that fall outside the windows.

B. Initial Calibration.

1. For the preparation of calibration blanks and calibration standards see Form 1−P−QM−FOR−9008921.

2. For the frequency, acceptance criteria and corrective action see tables I and II.

NOTE: The low standard must be at or below the LOQ.

C. Initial Calibration Verification (ICV).

1. For the preparation of ICV standard see Form 1-P-QM-FOR-9008921.

2. For the frequency, acceptance criteria and corrective action see tables I and II.

D. Continuing Calibration Verification (CCV).

1. For the preparation of CCV standard see Form 1-P-QM-FOR-9008921.

2. For the frequency, acceptance criteria and corrective action see tables I and II.

E. Low Level Check Standard (CRA)

1. For the preparation and concentrations of CRA standard see Form 1-P-QM-FOR-9008921.

2. For the frequency, acceptance criteria and corrective action see tables I and II.

Procedure

A. Sample preparation

1. Aqueous samples are digested according to Analysis #5713, 5714.

2. Solid samples are digested according to Analysis #5711, 10638.

B. Leeman Labs Hydra II Mercury Analyzer

1. Instrument Setup

a. Turn ON the power to the instrument (switch in the back) and computer.

b. Ensure that Argon supply is set to 15 psi.

c. Double click the Envoy icon on the desktop to initialize the instrument software.

d. Loosen all the peristaltic pump cassettes.

e. Place levers in the 1 o-clock position to avoid stalling the pump.

f. Check that the rinse bottle is full and Luer connections are tight. Only a 'light' finger tightening is required. Refill the rinse tank with a 2.0% Hydrochloric Acid (HCl) solution. For preparation of 2.0% HCl solution, see Form1−P−QM−FOR−9008921.

g. Check that the 10% stannous chloride bottle is full and Luer connections are tight. Only a 'light' finger tightening is required. For preparation of 10% stannous chloride, see Form 1−P−QM−FOR−9008921.

h. Click the start icon on the Tool Bar to turn on the peristaltic pump and set the gas flow to method programmed conditions.

I. Check to see that the lamp, pump and gas turn on. If necessary, open the Method/Instrument Control Panel and turn them on and set appropriate parameters.

j. When the pump is turning, tighten the cassettes by lowering the levers to a horizontal position. Allow 10 minutes for lamp and pump equilibration.

k. Inspect all system connections for leaks.

l. The system is now ready to be optimized for automated analysis.

2. Autosampler and Run Setup

a. Click on the Sequence Tab to display the automated sequence page.

b. Click the Sequence menu item on the Menu bar and select "Create" from the displayed options to display a spreadsheet of empty locations consisting of 3 racks with sample locations equal to the rack capacity (24, 60 or 90).

(1) Each row represents a cup location on one of the racks and its graphical representation updates in the lower "Rack" graphic whenever the Update button is clicked.

(2) Enter only laboratory sample numbers into the sample list table. The standards and Quality Control samples are automatically populated.

(3) Click the "Update" button to when all samples are entered to populate the navigation tree to the left with the proposed run sequence.

Sample Analysis

A. Leeman Labs Hydra II Mercury Analyzer

1. Analysis of samples with Leeman Labs Hydra II Mercury Analyzer

a. **Prior to analysis:**

(1) For soils remove cover, add 6 mL of sodium chloride/hydroxylamine hydrochloride solution to reduce excess permanganate. Adjust volume to the 100 mL mark with reagent water, and mix.

(2) For waters remove cover, add 2.4 mL of sodium chloride/hydroxylamine hydrochloride solution to reduce excess permanganate. Adjust volume to the 40 mL mark with reagent water, and mix.

b. Click the “Run Sequence” icon to start the run. If a dialog appears after the Run Sequence icon is clicked, follow the instructions of those prompts to resolve issues before running the sequence.

c. The system adds Stannous chloride to the samples via a “Y” connection in the pump tubing. The peristaltic pump then carries the sample/stannous mix to the liquid gas separator. Argon gas is bubbled through the liquid and used to transport the volatile mercury into the detector. The mercury is reduced with stannous chloride to liberate mercury metal and Tin (IV) chloride.

NOTE: Detailed instructions for the complete instrument setup are found in the *Leeman Labs Hydra II Automated Mercury Analyzer Manual*.

2. Dilutions

a. Dilute samples when necessary to yield a response that falls within the calibration range.

b. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

3. Instrument shutdown and cleanup

a. Overnight Shutdown

(1) Click on the “sleep” icon to stop argon flow and pump.

(2) In sleep mode the pump is cycled on periodically to relieve pressure points where the rollers contact the tubing.

(3) Never leave bottle of reductant and rinse connected to the instrument if the pump clamps are released because siphoning can occur and cause damage to the instrument.

b. Long-term shutdown (more than 3 days of no operation).

(1) Place reductant tubing and rinse tubing into a beaker of reagent water.

(2) Run pump until system is flushed of reagents. Send autosampler tip to air.

(3) Remove reductant and rinse tubing from beaker to allow the aspirating of air. Run pump until system is flushed of liquid. Some liquid does remain in the liquid/gas separator.

(4) Turn OFF the pump.

(5) Close Envoy program and power down the instrument.

(6) Shut OFF the power to the computer and monitor.

(7) Shut down argon gas flow.

4. Maintenance

a. Replace the pump tubing as needed under normal daily usage.

b. On an as needed basis, check the optical cell and windows, and if needed, clean the optical cell.

(1) Wipe the optical cell with a soapy solution (one drop of liquid Ivory soap to 500 mL reagent water) and warm tap water.

(2) Rinse with reagent water and dry. To speed the drying of the optical cell, connect the heater plug to the optical cell with the windows off for several minutes.

(3) Clean the quartz windows with methanol and a piece of lens paper.

(4) Document any maintenance in the Mercury maintenance logbook located next to the instrument.

NOTE: Detailed instructions for the maintenance and troubleshooting of the Leeman Labs Mercury Analyzer can be found in the *Leeman Labs Hydra II Mercury Analyzer Manual.*

Calculations

1. Final Result

a. Water sample

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

All dilution factors must be recorded and used in the calculation. [To enter dilution data into the LIMS when multiple dilutions are used, a factor must be formed (Ex. 1), which contains no more than three figures for the volume or the aliquot (Ex. 2).]

Ex. 1. 50/.5 × 10/1 = 500/.5

Ex. 2. 50/.5 × 25/.5 = 1250/.25 = 125/.025

NOTE: The default units are µg/L

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{N}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{N}\right)}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\%\ Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)

Take 4 identical aliquots of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution. Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^2 - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{\left[\sum (x_i - \bar{x})^2\right]\left[\sum (y_i - \bar{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ≥0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

Statistical Information/Method Performance

Generate MDLs and LOQs according to 1-P-QM-QMA-9017309. Perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire digestion and analysis procedure. Compare and pool results to determine the final reporting MDL. The department supervisor maintains annual study data. The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

Quality Assurance/Quality Control

A. For 7470A, 7471A, and 7471B, each digestion batch (up to 20 samples) must contain a method blank, LCS, and either an US, D, MS, MSD or an LCS/LCSD.

B. For 245.1, each digestion batch (up to 10 samples) must contain a method blank, LCS, and either an US, D, MS or an LCS/LCSD.

C. QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data.

1. The limits are maintained in the LIMS for the relevant analysis numbers.

2. The limits are evaluated every 6 months and updated as needed.

3. The limits are subject to change without notification.

D. Batch Quality Control

1. For the preparation and concentrations of Batch Quality Control see Form 1-P-QM-FOR-9008921.

2. For the frequency, acceptance criteria and corrective action see tables I and II.

E. Raw data quality checks

1. Confirm that the batch and cover sheets are correctly labeled, dated, and signed where necessary. Review the batch sheet, project notes and lab notes with the incomplete list for special comments and due dates. Check that the run protocol has been selected correctly.

2. Refer to the calculation section of this SOP for calculations used for Hg analysis.

3. Refer to Tables I and II for run and batch calibration and QC frequency, acceptance criteria and corrective action. For information on statistical limits see 1-P-QM-QMA-9017313.

4. Each analytical run must have a QC review attached. All samples on the run must be listed on the QC review with notation as to whether the sample was verified or needed to be redigested/reanalyzed. The verifier must document on the QC review if any sample(s) were selected/deselected.

5. For spike levels of run and batch QC, see Form 1-P-QM-FOR-9008921.

6. LOQs are available to analysts in the LIMS.

7. Check to make sure that all results are within the calibrations range. If a sample reading is above the calibration range, then reread the sample at an appropriate dilution.

8. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings ≤LOQ.

9. For TCLP and SPLP samples, an MSA (method of standard additions) is required if:

   a. The sample concentration falls between 80% to 100% of the regulatory limits.

   b. If the TCLP and SPLP matrix spike (QA) recovers <20%, all samples in the leachate batch must be reanalyzed using the method of standard additions for that analyte.

10. For all EW samples (samples from public drinking water sources); check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once so that the supplier can be notified. The verifier must contact the Client Service Representative, who must then notify the Supplier. Suppliers must be notified within 24 hours.

Analyte	MCL (mg/L)
Hg	0.002

F. When raw data checks are complete, check the following:

1. All samples requiring redigestion are listed on the redigestion schedule.

2. Redigest request forms are clipped to the front of the run.

3. The data are uploaded to eLIMS-EP via IDAT by reviewer then verified from eLIMS-EP by a verifier.

5. The data packet is placed in the verification bin.

G. Instrument detection limits are performed on a quarterly basis and method detection limits are performed on a yearly basis for each analytical instrument.

H. Taking an instrument/analysis out of service/returning an instrument/analysis to service.

NOTE: The following information is taken from 1−P−QM−QMA−9017325. In the event of an equipment failure, perform the following steps:

1. Document the nature of the failure in the maintenance logbook.

2. Document how and when the defect was discovered.

3. Notify a supervisor or experienced analyst to determine a person who can decide on appropriate action to take.

4. The instrument must be clearly tagged as *Out of Service*. The tag must contain the following information:

    a. Date taken out of service.

    b. Employee who took the instrument out of service.

    c. Reason for tagout.

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken and the result of that corrective action (i.e., system calibration within specifications, etc.) to bring the equipment back into service.

7. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument.

8. After a repair, document whether the function has been fixed. Calibration or verification activities are to be performed before the instrumentation is put back into service.

I. Verification process

1. Confirm that all required pieces of QC have been uploaded to eLIMS-EP and are within specification. If there is partial QC on the current run and the samples have been analyzed more than once, check to see if there are associated runs in the hold bin waiting on additional QC to be verified.

2. In eLIMS-EP, choose method of verification. (Metals verification by run or verify by individual element).

3. Ensure that all lab notes and project notes were followed.

4. Non-compliant data can be reported only after all required corrective actions have been taken. Document the nonconformance using Form 1-P-QM-FOR-9007858.

5. Once all of the elements are verified for a digest, verify the digest number. Associated tracking numbers or suite numbers will be auto-verified after all of the metals are verified.

Table 1

QC Requirements for EPA600 245.1 (Mercury) for (PW, EW) and (WW)

	Frequency	**Acceptance**	**Corrective Action**
Calibration	The calibration contains a blank and 5 standards. Due to the instrument software limitations, the calibration blank must be included in the correlation coefficient calculation.	Correlation coefficient >0.995	If the correlation coefficient is not met, confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the curve, if the correlation coefficient is acceptable, proceed with sample analysis. If the correlation coefficient is not met after reanalysis, redigest and reanalyze the curve and all associated samples.
Initial Calibration Verification (ICV)	Must be analyzed immediately following the calibration.	±5% of True Value	If the ICV is out of specification high report the elements that are < LOQ. For elements > LOQ, data cannot be reported. Confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the ICV, if the recovery is acceptable, proceed with sample analysis. If the acceptance criteria are not met after reanalysis, redigest and reanalyze the curve and all associated samples.
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV	Must be <ILOQI	Data cannot be reported from the run (reanalyze).
		±50% of the true value	

Contract Required Detection Limit (CRA)	Must be analyzed immediately after the ICB		Data cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the CRA and at the frequency of every 10 samples.	±10% of the true value	If the CCV is out of specification high and the sample is not < - LOQ, accept samples that report as non-detect. Data bracketing the CCV cannot be reported from other samples on the run (reanalyze). If the CCV is out of specification, it is read in duplicate. If both CCVs are within specification, the data from the last good CCV is reanalyzed. If one or both CCVs are still out of specification, then the run is terminated and the samples after the last good CCV must be analyzed on a new run.
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCVs at a frequency of every 10 samples.	Must be <ILOQI	Data bracketing the CCB cannot be reported from the run (reanalyze) If the CCB is out of specification, it can be read in duplicate. If both CCBs are within specification, the data from the last good CCB is reanalyzed. If one or both CCBs are still out of specification, then the run is terminated and the samples after the last good CCB must be reanalyzed on a new run.

	Frequency	**Acceptance**	**Corrective Action**
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	IPBI must be < ½ LOQ or 2.2x MDL whichever is greater. Not applicable if analyte reading in the sample is >10× the PB reading or < LOQ. EW samples are not accepted for any reason if the PB is out of specification.	Redigest all associated samples. EW samples must be redigested.
Laboratory Control Standard (LCS)	Must be prepped at a frequency if 1 per analytical batch of 10 samples or less. *Note: The LCS is spiked at or below the MCL for drinking water. See QA/QC section E.10. for MCL level.	Use statistical limits, or the method limit of ±15%, as indicated by the client requirement. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken. EW samples are not accepted if the LCS is out of specification.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high, redigest samples that are greater than the LOQ. EW samples must be redigested.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 10 samples or less. *Note: The LCS is spiked at or below the MCL for drinking water. See QA/QC section E.10. for MCL level.	Use statistical limits, or the method limit of ±15% As indicated by the client requirement. If the LCSD is out of specification high and the sample result is less than the LOQ the data can be taken. EW samples are not accepted if the LCS is out of specification. RPD must be <20%.	Redigest all associated samples if the LCSD is out of specification low. If the LCSD is out of specification high, redigest samples that are greater than the LOQ. EW samples must be redigested. Redigest if RPD is out of specification

Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	Use statistical limits or the method limit of ±30% whichever is tighter. (PW,EW) Use statistical limits or the method limit of ±20% whichever is tighter (WW)	The data is flagged in the QC Summary and/or in the data package.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	If the samples are >5× the LOQ the RPD must be <20%. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ.	The data is flagged in the QC Summary and/or in the data package.

Table II

QC Requirement for EPA SW846 7470A, 7471A and 7471B (Mercury)

	Frequency	**Acceptance**	**Corrective Action**
Calibration	The calibration contains a blank and 5 standards. Due to the instrument software limitations, the calibration blank must be included in the correlation coefficient calculation.	Correlation coefficient >0.995	If the correlation coefficient is not met, confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the curve, if the correlation coefficient is acceptable, proceed with sample analysis. If the correlation coefficient is not met after reanalysis, redigest and reanalyze the curve and all associated samples.
Initial Calibration Verification (ICV)	Must be analyzed immediately following the calibration.	±10% of True Value	If the ICV is out of specification high report the elements that are < LOQ. For elements > LOQ, data cannot be reported. Confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the ICV, if the recovery is acceptable, proceed with sample analysis. If the acceptance criteria are not met after reanalysis, redigest and reanalyze the curve and all associated samples.
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV	Must be <ILOQI	Data cannot be reported from the run (reanalyze).
Contract Required Detection Limit (CRA) Limit of Quantitation Check Standard	Must be analyzed immediately after the ICB	For 7470A and 7471A: ±50% of the true value For 7471B: 30% of the True Value	Data cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the CRA and at the frequency of every 10 samples.	±20% of the true value	If the CCV is out of specification high and the sample is not < - LOQ accept elements that report as non-detect. Data bracketing the CCV cannot be reported from other samples on the run (reanalyze) If the CCV is out of specification, it is read in duplicate. If both CCVs are within specification, the data from the last good CCV is reanalyzed. If one or both CCVs are still out of specification, then the run is terminated and the samples after the last

			good CCV must be reanalyzed on a new run or redigested if there is not enough CCV (or any other run standard) to reanalyze with a new calibration.
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCVs at a frequency of every 10 samples.	Must be <ILOQI	Data bracketing the CCB cannot be reported from the run (reanalyze) If the CCB is out of specification, it is read in duplicate. If both CCBs are within specification, the data from the last good CCB is reanalyzed. If one or both CCBs are still out of specification, then the run is terminated and the samples after the last good CCB must be reanalyzed on a new run or redigested if there is not enough CCB (or any other run standard) to reanalyze with a new calibration

	Frequency	**Acceptance**	**Corrective Action**
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	IPBI must be < ½ LOQ . For 7470A and 7471A: Not applicable if analyte reading in the sample is >20× the PB reading or <LOQ. For 7470B and 7471B: Not applicable if analyte reading in the sample is >10× the PB reading or <LOQ.	Redigest all associated samples.
Laboratory Control Standard (LCS)	Must be prepped at a frequency if 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high, redigest samples that are greater than the LOQ.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement. If the LCSD is out of specification high and the sample result is less than the LOQ the data can be taken. RPD must be <20%.	Redigest all associated samples if the LCSD is out of specification low. If the LCSD is out of specification high, redigest samples that are greater than the LOQ. Redigest if RPD is out of specification
Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20% whichever is tighter.	The data is flagged in the QC Summary and/or in the data package.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	If the samples are >5× the LOQ the RPD must be <20%. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ.	The data is flagged in the QC Summary and/or in the data package.

End of document

eurofins	Always check on-line for validity **Mercury in Aqueous, Solid and Tissue Samples by Cold Vapor AA**	Level: **Work Instruction**
Document number: **T-MET-WI7965**		
Old Reference: **1-P-QM-WI-9015067**		
Version: **16**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **27-NOV-2015**	Document users: **6_EUUSLA_Metals_Hg Analysis, 6_EUUSLA_Metals_Hg Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

Version history

Version	Approval	Revision information
16	27.NOV.2015	

eurofins	Always check on-line for validity	Level:
Document number: **T-MET-WI11924**	**Digestion of Aqueous Samples by SW-846 Method 7470A, EPA 245.1**	**Work Instruction**
Old Reference: **1-P-QM-WI-9015082**		
Version: **19.1**		Organisation level: **5-Sub-BU**
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LIMS ID
Analysis DOD - 5713, 5714

Revision Log

Revision: 19		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Correction of transcription error and clarification	Change EPA 254.1 to EPA 245.1 and add mercury analysis
Table of Contents	Unnecessary section	Removed section
Cross Reference	Higher level documents are not required to be referenced	Removed 1-P-QM-QMA-9015390
Purpose	Information contained within the scope	Removed section
Definitions	Optional section deemed unnecessary	Removed section

Revision: 19	**Effective Date:** This version	
Personnel Training and Qualifications	Higher level documents are not required to be referenced	Removed 1-P-QM-QMA-9015390
Apparatus and Equipment	Enhancement	Moved LLENS into this section from the definitions
Procedure 6	Reflects current procedure	Added text pertaining to spiked leachate QA samples.

Revision: 18	**Effective Date:** Apr 29, 2015	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	No longer part of current procedure	Deleted CLP2.1, CLP4.0, and CLP5.2
Historical/Local Document Number	No longer part of current procedure	Deleted 0821
Throughout Document	No longer part of current procedure No longer used for this procedure	Deleted reference to CLP2.1, CLP4.0, and CLP5.2 Deleted reference to the use of DEENA Automated Sample Preparation System
Cross Reference	Reflect current procedure	Added 1-P-QM-QMA-9015390
Safety Precautions and Waste Handling	Clarification	Added the use of designated dispensing equipment for acids. Added text pertaining to hazardous flags.
Personnel Training and Qualifications	Reflect current procedure	Added 1-P-QM-QMA-9015390
Sample Collection, Preservation, and Handling	Clarification	Added text pertaining to pH checks and pH adjustments.
Apparatus and Equipment	No longer used for this procedure	Deleted 100 mL polypropylene containers
Reagents and Standards	Clarification No longer used for this procedure	Added reference to 1-P-QM-FOR-9008921 for reagent preparation, shelf life, and storage conditions. Deleted NOTE referencing the use of water bath.
Reagents and Standards C.	Reflect current procedure	Deleted preparation of Calibration Curve, ICV, CCV, CRA, ICB, and CCB
Block Digester Instructions	Reflect current procedure	Added entire section

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 7470A, September 1994

2. Method 245.1 (rev. 3), Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectroscopy, USEPA 600/R-94/111 May 1994.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
1-P-QM-FOR-9008921	Working Instructions for Preparation of Mercury Solutions and Standards

Scope

This procedure is used for automated and manual digestion of samples to be analyzed for Mercury in aqueous samples by SW-846 Method 7470A and EPA 245.1.

Basic Principles

The samples are digested with nitric acid, sulfuric acid, potassium permanganate, and potassium persulfate to oxidize mercury compounds to mercuric ions. Mercuric ions are reduced to mercury metal using stannous chloride. Mercury measurement is performed using the mercury cold vapor technique.

The DEENA Automated Sample Preparation System utilizes automated addition of reagents, heating, and filling to final volume to digest Mercury samples.

Reference Modifications

Manual digestions: To increase efficiency, polypropylene containers are used in place of BOD bottles. Prior to analysis (after excess potassium permanganate is reduced with sodium chloride/hydroxylamine hydrochloride solution) samples are adjusted to 50 mL in volumetric flasks. This allows aliquots to be taken as required for analysis; aliquots cannot be taken when BOD bottles are used.

To increase efficiency and temperature accuracy, a hot block digester is used in place of a 95°C water bath.

Interferences

Not applicable to this procedure.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO_3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes, or use the designated dispensing equipment.

Sulfuric acid (H_2SO_4) – This acid is a strong oxidizing agent and can cause severe burns. Sulfuric acid spills are extremely slippery, adding to the danger. Always use in a fume hood, or use the desinated dispensing equipment. Never mix with concentrated HCl or concentrated KMNO4 to avoid a violent reaction (explosive splattering and extreme heat).

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H_2SO_4 to avoid a violent reaction. Always use in a fume hood, or use the designated dispensing equipment.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each employee performing the digestion must work with an experienced employee for a period of time until they can independently perform digestions. Proficiency is measured through documented audits of the tasks listed as well as an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (LCS's) that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Aqueous samples are collected in plastic or glass containers (drinking water sample volume is 1-L). Samples are preserved to a pH of <2 with nitric acid at collection and the pH is checked upon receipt and adjusted as necessary by Sample Support; samples that are pH adjusted at the laboratory must not be digested for a minimum of 24 hours. If samples fail to maintain a pH of <2 the Client Representative is notified for further direction. Samples are stored at 0 to 6°C, not frozen, prior to digestion. Samples must be digested within 28 days of collection.

Dissolved Mercury: Samples to be analyzed for soluble mercury requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt and then preserved to a pH of <2 with HNO_3.

Digested samples are stored in plastic bottles at room temperature. Store samples, standards, and digested samples separately.

Apparatus and Equipment

1. 50-mL polypropylene containers and covers (digestion vessels for block digestion) – certified clean and Class A equivalent

2. Environmental Express HotBlock (block digester) – adjustable and capable of maintaining a sample temperature of 95°C.

3. LLENS – the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the LIMS.

Reagents and Standards

A. For standards preparation, shelf life, and storage conditions, see Form 1−P−QM−FOR−9008921. This form also has instructions for the initial preparation of Method Blanks, LCSs, Matrix Spikes and General Solutions.

B. Store all standards and reagents at room temperature. Label the container with the solution name, lot number, date prepared, the expiration date, the initials of the person preparing the solution, and the storage conditions.

C. Standard/ spiking concentration and reagent vendors are subject to change without notification.

E. Nitric acid, 70.0% to 71.0% HNO_3, Fisher Trace Metal Grade reagent, 1.428 g/mL, or equivalent. Follow manufacturer's expiration date.

F. Sulfuric acid, 95.0% to 98.0%, H_2SO_4, 36 N, Fisher reagent, ACS, 1.84 g/mL, or equivalent. Follow manufacturer's expiration date.

G. Potassium permanganate, $KMnO_4$, Baker Analyzed reagent, ACS, or equivalent. Follow manufacturer's expiration date.

H. Potassium persulfate, $K_2S_2O_8$ Baker Analyzed reagent, ACS, or equivalent. Follow manufacturer's expiration date.

I. Sodium chloride, NaCl, J.T. Baker, Certified ACS, or equivalent. Follow manufacturer's expiration date.

J. Hydroxylamine hydrochloride, $NH_2OH{\bullet}HCl$, J.T. Baker, Certified ACS, or equivalent. Follow manufacturer's expiration date.

K. Stannous chloride, SnCl, Baker Analyzed reagent, ACS, or equivalent. Follow manufacturer's expiration date.

L. Hydrochloric acid, HCl, 36.5% to 38.0%, Fisher Trace Metal Grade reagent, 1.194 g/mL or equivalent. Follow manufacturer's expiration date.

Calibration

Not applicable to this procedure

Procedure

A. Manual hot block digestion

1. Turn block digester on and allow block to reach the Control Point setting that provides 95° ± 1°C sample temperature. The temperature is checked with a calibrated thermometer and recorded in the logbook.

2. Print labels with the LLI Sample ID and batch number from LLENS and place on sample digestion containers.

3. Shake sample well.

4. Transfer 40 mL of well−mixed sample (or an aliquot diluted to 40 mL) into the polypropylene container. If a different final volume is necessary, adjust reagent volumes accordingly.

5. See Form 1−P−QM−FOR−9008921 for instructions on preparing batch and instrument Quality Control, calibration and for concentration levels.

NOTE: Exception - For leachate batches containing leachate spiked QA samples. Use one of the leachate spiked QA samples as the batch QC matrix spike and matrix spike dup, and proceed with the spiking procedures for the LCS according to Form 1−P−QM−FOR−9008921.

6. Add 2 mL of H_2SO_4 and mix.

7. Add 1 mL of HNO_3 and mix.

8. Add 6 mL of 5% $KMnO_4$ solution and mix.

9. Allow the sample to stand for 15 minutes and then check sample for purple color.

a. If the purple color does not persist for at least 15 minutes, perform a dilution on the sample beginning with a DF5.

b. Continue diluting in increments of 5 or 10 until the purple color persists for at least 15 minutes.

10. Add 3.2 mL of 5% $K_2S_2O_8$ solution and mix.

11. Place containers in block digester.

12. Place a calibrated thermometer in method blank container.

13. Put a polypropylene cover on each container. Remove after the samples reach 95° ± 1°C.

14. Heat for 2 hours in the block digester at 95° ± 1°C. The temperature is checked with a calibrated thermometer and recorded in the logbook.

15. Remove samples from block digester and cool.

16. Screw on disposable cap.

17. Prior to analysis, add 2.4 mL of 12% sodium chloride/hydroxylamine hydrochloride solution to reduce excess permanganate (color change is from purple to colorless). If the solution is not colorless, add reductant in 1−mL increments until KMnO4 is completely reduced.

18. Adjust the volume to 40 mL with reagent water and mix. Reserve for analysis.

NOTE: The block temperature is different than the temperature of the liquid being digested.

B. Block Digester Instructions:

1. Turn block digester on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. Press and hold STAR (*) key.

4. The display shows the Set Point Temperature.

5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations
Not applicable

Statistical Information/Method Performance
Not applicable

Quality Assurance/Quality Control
A. For 7470A, each digestion batch is up to 20 samples.

B. For 245.1, each digestion batch is up to 10 samples.

C. Each digestion batch must contain a method blank, LCS, and either an US, D, MS or an LCS/LCSD.

1. Method Blank – equivalent to a Preparation Blank (PB).
2. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate
3. U or US – unspiked background sample
4. D – duplicate
5. MS (R) – Matrix Spike
6. MSD (M) – Matrix spike duplicate

End of document

Version history

Version	Approval	Revision information
19	13.OCT.2016	
19.1	22.MAY.2017	

eurofins	Always check on-line for validity **Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for Analysis of Metals by ICP and ICP-MS**	Level: **Work Instruction**
Document number: **T-MET-WI8636**		
Old Reference: **1-P-QM-WI-9015160**		
Version: **23**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **23-DEC-2015**	Document users: **6_EUUSLA_Metals_ICP Prep, 6_EUUSLA_Metals_ICP Prep Verifiers, 6_EUUSLA_Metals_ICP-MS Prep, 6_EUUSLA_Metals_ICP-MS Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis DOD - 5708, 10637

Revision Log

Revision: 23		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Clarification	Add method reference: Sample Prep of Sediment, Sludge, Soil, and Tissue by SW846 3050B for Analysis of Metals by ICP and ICP-MS
Personnel Training and Qualifications	Enhancement	Added information on DOC options and the SOP on Demonstrations for Capability

eurofins	Always check on-line for validity **Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for Analysis of Metals by ICP and ICP-MS**	Level: **Work Instruction**
Document number: **T-MET-WI8636**		
Old Reference: **1-P-QM-WI-9015160**		
Version: **23**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **23-DEC-2015**	Document users: **6_EUUSLA_Metals_ICP Prep, 6_EUUSLA_Metals_ICP Prep Verifiers, 6_EUUSLA_Metals_ICP-MS Prep, 6_EUUSLA_Metals_ICP-MS Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

Revision: 23	**Effective Date:**	This version
Procedure	Process change	Changed high end of initial weight from 1.05g to 1.50g.

Revision: 22	**Effective Date:**	Aug 21, 2014
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Clarification	Removed Fish from the title so that the word Tissue is not limited to only Fish.
Procedure	Clarification	Removed the word fish from tissue section and included or other tissue samples are used.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3050B, December 1996.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A (aqueous, solid, tissue) and EPA 200.8 (aqueous)
Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, …	Metals by Inductively Coupled Plasma Atomic Emissions Spectroscopy for SW-846 Methods 6010B/C (aqueous, solid, tissue) and EPA 200.7(aqueous)
1-P-QM-FOR-9009182	Working Instructions for Prep Solutions and Standards
1-P-QM-QMA-9015390	Demonstrations of Capability

Purpose

This digestion procedure is for the preparation of solid samples for analysis by ICP and ICP/MS following SW-846 protocol.

Scope

This method is used for preparation of metals in solid samples for analysis by ICP and ICP/MS.

Basic Principles

A representative sample is digested with repeated additions of nitric acid (HNO3) and hydrogen peroxide (H2O2). Hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed. The resultant digestate is diluted and analyzed.

This method is not a total digestion technique for most samples; it is a very strong acid digestion that dissolves almost all elements that could become “environmentally available.” By design, elements bound in silicate structures are not normally dissolved by this procedure.

Definitions

1. ACS – American Chemical Society

2. D – Sample Duplicate

3. DOC – Demonstration of Capability

4. IDOC – Initial Demonstration of Capability

5. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate

6. LCSW– Laboratory Control Sample Water

7. LLENS - the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the LIMS.

8. LIMS – Laboratory Information Management Systems

9. LLI Sample ID – unique 7-digit number assigned to a client sample.

10. LOQ – Limit of Quantitation

11. MDL – Method Detection Limit

12. MS (R) – Matrix Spike

13. MSD (M) – Matrix spike duplicate

14. PB/PBW–Preparation Blank/ Preparation Blank Water

15. QC – Quality Control

16. Method Blank - equivalent to a Preparation Blank. A designated sample designed to monitor for sample contamination during the analysis process. A volume of reagent laboratory water is typically

used to monitor water sample analysis, while solids blanks consist of a purified solid matrix or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

17. SOP- Standard Operating Procedure

18. U or US – unspiked background sample

Interferences

When analyzing sample by ICP-MS using this digestion procedure we follow the instrument manufacturer's guidelines to eliminate polyatomic interferences typically caused by Chlorine. The process we follow involves the use of a collision/reaction cell on the ICP-MS. Below is a description of how the collision/reaction cell works.

Reaction Process - The primary method of interference removal is through a reaction event. When using a reaction gas, either the target interference is more reactive than the target analyte, leading to preferential removal of the interferent or (less commonly) the target analyte is more reactive and is converted to a new species at a different mass which is free from any existing or newly-formed overlap

Collision Process - The primary method of interference removal is through a non-reactive event. This process of interference removal is kinetic energy discrimination (KED). Energy Discrimination is most commonly used with an inert gas, which means the interference removal process is not affected by reactions in the cell.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO3) – This acid can cause skin burns. Add nitric acid to samples in a hood or use the designated dispensing equipment to avoid exposure to toxic fumes.

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood or use the designated dispensing equipment.

Hydrogen peroxide (H2O2) - This is a strong oxidizing agent and causes severe burns. Avoid contact with skin.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documentation of the critical steps in this procedure, over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and

standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample. Refer to 1-P-QM-QMA-9015390 (Demonstrations of Capability) for more guidance on these options.

Sample Collection, Preservation, and Handling

Solid samples require no chemical preservation.

Samples must be submitted in glass or plastic containers and stored at 0° to 6°C, not frozen, prior to digestion. Samples must be digested within 6 months (180 days) of sample collection.

Digested samples are stored in polypropylene bottles at room temperature.

Apparatus and Equipment

1. Polypropylene containers and covers (digestion vessels) - certified clean and Class A equivalent

2. Whatman No. 41 filter paper or equivalent

3. Funnels

4. Environmental Express HotBlock (block digestor) - adjustable and capable of maintaining a temperature of 90 to 95°C

5. Balance capable of reading 0.01 g

6. Chemware Ultra-Pure PTFE boiling stones, or equivalent.

7. Computer and software LLENS (Lancaster Laboratories Electronic Notebook System)

Reagents and Standards

For reagent preparation, shelf life, and storage conditions, see Form 1-P-QM-FOR-9009182.

1. Nitric acid (HNO_3) – Fisher, Trace Metal Grade, or equivalent. Store at room temperature. Re-evaluate annually.

2. Nitric acid (1:1) – Add 500 mL of HNO_3 to 500 mL of reagent water. Store in polypropylene at room temperature. Expires 6 months from date of preparation. (Different volumes are acceptable but ratios must stay the same.)

3. Hydrogen peroxide, 30% (H_2O_2) – Fisher, Certified ACS or equivalent. Store at room temperature. Re-evaluate annually.

4. Hydrochloric acid (HCl) – Baker Instra-Analyzed, or equivalent. Store at room temperature. Re-evaluate annually.

NOTE: It is acceptable to prepare using multiples of indicated weights and volumes if ratios are maintained.

Calibration

Not applicable.

Procedure

1. Turn block digestor on and allow block to reach the Control Point setting that provides 90° to 95°C sample temperature. (The block temperature setting is not necessarily the sample temperature.) See below for **Block Digestor Instructions** section.

2. Weigh 1.00 to 1.50 g (to the nearest 0.01 g) of a well mixed sample into a polypropylene digestion vessel. (If the sample is watery use 5.00 to 5.50 grams for analysis. Additional information on non-standard matrices is found at the end of the procedure section.) Add 1.00 to 1.49 g of Chemware Ultra-Pure PTFE boiling stones to the digestion vessel for the blank and LCS. Enter the blank and LCS weight as 1.0000 to 100.0000 final volume in the LLENS. For sample batch spiking procedures see 1−P−QM−FOR−9009182. All spiking must be performed prior to starting the digestion procedure.

3. Add 10 mL of (1:1) HNO_3, swirl to mix, and cover with a polypropylene cover.

4. Place sample vessel in block digestor. Heat (reflux) the sample at 90° to 95°C for 10 to 15 minutes without boiling.

5. Remove vessel from digestion block and allow sample to cool.

6. Add 5 mL of concentrated HNO_3. Replace cover, return vessel to digestion block and heat for 30 minutes.

NOTE: If brown fumes are generated (indicating oxidation of the sample by HNO_3) continue the process of adding 5 mL HNO3 and heating until no brown fumes are given off by the sample. This indicates that the reaction with HNO_3 is complete. Add the same amount of HNO_3 to the entire digestion batch.

7. With cover on, heat at 90° to 95°C without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times (add reagent water if necessary).

8. Remove vessel from digestion block and allow sample to cool.

9. Add 2 mL of reagent water and 3 mL of 30% H_2O_2. With cover on, return vessel to digestion block and heat until effervescence subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

10. Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

NOTE: Do not add more than a total of 10 mL 30% H_2O_2.

11. With cover on, continue heating the acid-peroxide digestate at 90° to 95°C without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times (add reagent water if necessary).

12. Remove sample vessel from digestion block and allow to cool.

13. Add 10 mL of HCl. With the cover on, return vessel to digestion block and heat at 90° to 95°C for 15 minutes.

14. Remove sample vessel from digestion block.

15. If floating particulate is evident after digestion, the sample must be filtered.

   a. Filter through Whatman No. 41 filter paper into a polypropylene container.

   b. Wash sample vessel, residue, and paper thoroughly with reagent water.

   c. If any samples are filtered, the prep blank and LCS must also be filtered.

16. Adjust volume to the 100mL mark on the digestion vessel with reagent water and mix. Seal vessel with a screw cap. The sample is now ready for analysis.

NOTE: When special limits of quantitation are required by the client, use more sample weight.

<u>For wipe samples</u>:

When wipes are digested by this method, one blank media each must be used for the batch preparation blank, the LCS, and the LCSD. Refer to Form 1-P-QM-FOR-9009182 for the spiking of the LCS and LCSD. Digest wipes in their own batch. Use reagent water to rinse any particulate matter from the wipe container into the vessel containing the wipe before digesting. If brown fumes are evolved during wipe sample digestion, perform only two 5 mL HNO_3 additions with 30-minute refluxing each; add the same amount of HNO_3 to the entire batch. Proceed with digestion.

For tissue samples:

When fish tissues, or other tissue samples are digested by this method, refer to Form 1-P-QM-FOR-9009182 for the spiking of the LCS, LCSD (if needed), R (matrix spike), and M (matrix spike duplicate). Add 1.00 to 1.49 g of Chemware Ultra-Pure PTFE boiling stones to the digestion vessel for the blank and LCS. Digest tissue samples in their own batch.

Block Digestor Instructions

1. Turn block digestor on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. PRESS and hold STAR (*) key.

4. The display shows Control Point temperature.

5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 90° to 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used. If necessary, adjust Control Point temperature to the proper setting as instructed below.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations

Not applicable.

Statistical Information/Method Performance

Not applicable to this procedure. See analysis method.

Quality Assurance/Quality Control

For sample batch spiking instructions see form 1-P-QM-FOR-9009182. Refer to ICP section when prepping ICP analysis. Refer to ICP/MS section when prepping ICP/MS analysis. Prepare a method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample with every digestion batch (20 samples or less). Each piece of batch QC is digested following the procedure in this SOP. If any samples are filtered the prep blank and LCS must also be filtered.

Refer to ICP Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961, 10145, 6955, 6944, 6936, 6969, 7968, … for sample batch quality control requirements, acceptance criteria and corrective action.

Refer to ICP/MS Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, … for sample batch quality control requirements, acceptance criteria and corrective action.

End of document

Version history

Version	Approval	Revision information
23	23.DEC.2015	

eurofins	Always check on-line for validity **Microwave Extraction Method 3546 for PCBs in a Solid Matrix**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10927**		
Old Reference: **1-P-QM-WI -9015104**		
Version: **7**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **03-DEC-2015**	Document users: **5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Pest/PCB Soils, 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID
Analysis DOD - 10497, 11140, 13100

Revision Log

Revision: 7	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P -QM-QMA-9017356	Removed revision logs up to the previous version
Historical/Local Document Number	Added new analysis to procedure	Add 13100 to historical document number.
Procedure	Updated to current process	Added information about sample aliquot

Revision: 6	**Effective Date:** Sep 29, 2014

Revision: 6	**Effective Date:** Sep 29, 2014	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Document Title	Enhancement	Added Method 3546
Cross Reference	Deactivated	Removed analysis 1216, 5108, 2033, 10885.
Sample Collection, Preservation, and Handling	Reflects current industry standards	Updated refrigeration conditions from 4º ± 2ºC.
Reagents and Standards 5.	Deactivated LIMS scans	Removed analysis 1216, 5108, 2033, and 10885.
Procedure 8.	Enhancement	Added information on temperature of microwave and documentation

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3546, Revision 0, February 2007.

2. *Chemical Hygiene Plan*, current version.

3. MARS Operation Manual, Revision 2, February 2006.

Cross Reference

Document	Document Title
Analysis #0042, 1030, 6011, 7512, 10225, 10736, 10906, 12800, 13236	Polychlorinated Biphenyls (PCBs) by Method 8082 in Solids and Wipes
Analysis #2487	Food and Tissue Preparation
1-P-QM-PRO-9015407	Pesticide Extract Cleanup Using Gel Permeation Chromatography
1-P-QM-PRO-9015475	Glassware Cleaning for Organic Extractions
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Purpose

The purpose of this SOP is to clearly outline the steps taken to extract solid samples for analysis of PCB compounds using microwave technology.

Scope
This procedure is applicable for the extraction of PCBs from soils or solid wastes.

Basic Principles
A portion of sample to be analyzed is placed in an extraction vessel. Surrogate standards are added to each sample to monitor recovery. The vessel is then loaded into the microwave and extracted. The organic compounds present in the sample dissolve in the solvent, which is then removed. The sample is then concentrated and bottled.

Several cleanup procedures may be required to eliminate matrix interferences before the sample can be analyzed. They include: sulfuric acid treatment, florisil, copper, and gel-permeation cleanup (GPC).

Reference Modifications
The KD is not removed from the steam bath and cooled before adding hexane. The Snyder column is not removed for the solvent addition; therefore, the KD does not have to be cool.

Double volumes of surrogates and matrix spiking solutions are not added when a sample requires gel-permeation cleanup. Instead, the extract is concentrated to one-half the normal final volume after GPC to make up for the loss on GPC and maintain the limit of quantitation.

The Joint of the K-D is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

Definitions
1. GPC – Gel Permeation Cleanup

2. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – A sample of known composition analyzed with each batch of samples to demonstrate laboratory accuracy. The samples either naturally contain the analytes of interest or are clean samples fortified with known concentrations. Used to demonstrate laboratory accuracy. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

3. Matrix spike/matrix spike duplicate (MS/MSD) – A sample created by fortifying a second aliquot of a water or soil sample with some or all of the analytes of interest. The concentration added is known and compared to the amount recovered to determine percent recovery. Matrix spike recoveries provide information about the accuracy of the method in light of the matrix analyzed. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

4. Surrogates – Organic compounds which are similar to the analytes of interest but are not naturally occurring in environmental samples. Surrogates are spiked into all standards and every field and QC sample prior to extraction and analysis to provide information regarding the effects of the sample

Interferences

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware is rinsed with solvent before use and a method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.
Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or must be disposed of in the designated containers. These will then be transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the technicians training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass jars with PTFE-lined lids and stored under refrigeration at 0º to 6º C, not frozen, prior to extraction. Samples must be extracted within 14 days of collection. Extracts are stored in the freezer at -10° to -15°C.

Apparatus and Equipment

1. MARS Xpress – CEM Corp. or equivalent

2. Kuderna-Danish (K-D) assembly with appropriate ampule for concentrating the solvent used during concentration

3. Steam bath, VWR/LLI Model #1127 or equivalent

4. N-Evap with nitrogen supply

5. Beakers – Stainless steel, assorted sizes

6. Pipettes – Class A, assorted sizes

7. Graduated cylinders – Class A, assorted sizes

8. Pipettes – Disposable

9. Balance – Capable of weighing to 0.01 g

10. Teflon®-wash bottles

11. Vials – Assorted sizes

12. Teflon®-boiling chips

13. Forceps

14. Scoop

15. TurboVap II concentration workstation w/appropriate concentration tubes – Zymark or equivalent

16. Funnels – stainless steel or Teflon®

17. xtraction vessels

18. Frits – Various

19. Sodium Sulfate Columns

Reagents and Standards

1. Hexane – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt

2. Acetone – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt

3. Methylene Chloride (CH_2Cl_2) – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt

4. Sodium Sulfate (Na_2SO_4) – Reagent grade or equivalent. Bake at 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

5. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding Analysis #0042, 1030, 6011, 7512, 10225, 10736, 10906, 12800, 13236.

Preparation of Glassware

See 1-P-QM-PRO-9015475 (SOP-OE-001).

Calibration

Not applicable to this procedure

Procedure

1. Sample aliquot

a. If Sample Registration has pre-weighed the sample into a glass jar, add 5g of sodium sulfate, mix and proceed.

b. If the sample is not pre-weighed, weigh 30.0 - 30.5 g of sample into a labeled stainless steel beaker.

(1) Record the initial weight and any comments about the sample in the extraction log.

(2) Alternative sample weights may be used to meet certain reporting limits. However, if sample weight <30.5 is used the sample must be divided among multiple vessels and the extracts combined.

c. Process all tissues by Analysis #2487 prior to extraction.

d. Add 5 g of sodium sulfate to each sample and mix.

(1) If the sample has high water content or is a clay-like soil, add an additional 10 g of sodium sulfate.

(2) Mix the sodium sulfate and sample until a free-flowing consistency is reached.

e. The Blank, LCS, and LCSD (if applicable) are prepared by filling a Teflon® extraction vessel with 35.0 g of sodium sulfate.

Record 30.0 g on the extraction log. (The sodium sulfate used for the QC samples is measured out as 35.0 grams to account for the 30 gram "sample" plus the 5 grams of sodium sulfate.)

2. Carefully place each sample into its clearly marked corresponding extraction vessel. A funnel is be used to prevent spillage and loss of sample.

3. Use pipettes to add surrogate standards and spiking solutions.

a. Surrogates – 1.0 mL of SW846 surrogate is added to all samples (including QC).

b. Spiking solutions – 1.0 mL of PCB spiking solution is added to the LCS, LCSD if applicable, MS, and MSD.

c. This may change to accommodate specific client requirements.

d. See 1-P-QM-PRO-9015490 (SOP-OE-017) for storage and handling of spikes.

4. Add 30 mL of 50% acetone in methylene chloride to each vessel.

5. Cap each vessel according to manufacturer's guidelines.

6. Invert each vessel to ensure mixing of sample and solvent.

7. Place the vessels into the carousel. When all samples are loaded, place the carousel into the microwave.

8. Run Program "LL1". See Table I for Instrument conditions. Verify that the run reached 100°C and document on the batchlog.

9. Uncap the cooled vessel. Pour the extract and sample into a column filled with approximately 10cm of sodium sulfate on top of a Kuderna-Danish (K-D) assembly containing a Teflon®-boiling chip. Rinse the vessel with 10-20 mL of hexane from a wash bottle.

10. Place a 3-ball Snyder column on the K-D set-up, wet the column with hexane, and concentrate over a steam bath which is at 85° to 99°C.

This steam bath temperature ensures concentration in a reasonable length of time.

If the sample requires GPC, skip step 11.

11. When the apparent volume is 3 to 5 mL, using a graduated cylinder, add approximately 50 mL of hexane directly to the KD through the Snyder column.

Do not allow the ampule to go dry since loss of analytes will result.

12. When the apparent volume again reaches 3 to 5 mL, remove from the steam bath and allow to cool for 10 minutes. Remove the ampule and using a wash bottle adjust the final volume to exactly 10 mL with hexane in a calibrated ampule.

NOTE: If the sample requires GPC, N-Evap to approximately 1 mL, then adjust the final volume to exactly 10 mL with methylene chloride instead of hexane. N-Evap if necessary.

Mix thoroughly with a disposable pipette.

13. If the sample requires GPC, perform GPC cleanup following 1-P-QM-PRO-9015407 (MC-OE-004). When GPC cleanup is complete, concentrate the extract as described above including Step 11. However, in Step 12, adjust the final volume to exactly 5 mL with hexane instead of 10 mL.

14. Treat the 10 mL extract with sulfuric acid as described in 1-P-QM-PRO-9015477 (SOP-OE-004).

15. Florisil the extract following the PCB Florisil section of 1-P-QM-PRO-9015477

16. Perform copper cleanup of the extract as described in 1-P-QM-PRO-9015477

17. Bottle twice in an appropriately labeled crimp-top autosampler vial, and place the remaining extract in an appropriately labeled screw-cap vial.

18. All extracts are stored in the freezer.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
A batch is defined as the samples to be extracted in any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

For each batch of samples extracted, a blank, LCS, MS, and MSD must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Table I

Power:	1600W
Ramp Temperature:	100°C
Ramp Time:	30 minutes
Hold Time:	10 minutes
Cool Down Time:	20 minutes

End of document

Version history

Version	Approval	Revision information
7	03.DEC.2015	

An error has happened on file: /D4Doc/formularer/2016/09/Quality Document # 1-P-QM-WI -9015104_7_1049711140.doc
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eurofins	Always check on-line for validity **Polychlorinated Biphenyls (PCBs) in Solid Samples by 8082A Using GC-ECD**	Level: **Work Instruction**
Document number: **T-PEST-WI10004**		
Old Reference: **1-P-QM-WI-9015110**		
Version: **8**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **26-AUG-2016**	Document users: **6_EUUSLA_Pesticide Residue Analysis_All Chemist Tr, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Pesticide Residue Analysis_Manager**

LIMS ID

Analysis DOD - 10592, 10885, 12718, 13099, 13219, 13713

Revision Log

Revision: 8		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Cross Reference	References not needed	Removed 1-P-QM-QMA-9015390 and 1-P-QM-QMA-9017309
Scope	Reflects current Aroclors	Updated table to include Aroclor 1262 and 1268 Added 1262 and 1268 to analysis scan 13099 in the table

eurofins	Always check on-line for validity **Polychlorinated Biphenyls (PCBs) in Solid Samples by 8082A Using GC-ECD**	Level: **Work Instruction**
Document number: **T-PEST-WI10004**		
Old Reference: **1-P-QM-WI-9015110**		
Version: **8**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **26-AUG-2016**	Document users: **6_EUUSLA_Pesticide Residue Analysis_All Chemist Tr, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Pesticide Residue Analysis_Manager**

Revision: 8	**Effective Date:**	This version
Basic Principles	Old reference not needed	Removed old reference number SOP-OE-004
Reference Modification	Reflects the current reference method	Updated reference to 8082A
Definition	Common laboratory terms defined in higher level documents	Removed definitions
Interferences	Old reference not needed.	Removed old reference number SOP-OE-004
Personnel Training and Qualifications	Reference not needed	Removed reference 1-P-QM-QMA-9015390.
Sample Collection, Preservation, and Handling	Reflects current storage condition for extracts	Added temperature storage condition for extracts.
Apparatus and Equipment	Reflects current instrumentation in use	Updated to HP7890
Reagents and Standards	Reflects current standard solution storage condition	Updated standard solution temperature storage condition from -10 to -15°C to ≤-10°C
Reagents and Standards 18.d	Reflects current MS and SS spike solutions	Updated to the current spike solution names.
Extraction	Reflects current scans	Added Analysis #13100
Calibration	Reflects current process Reflects current analysis sequence Reflects current naming convention	Removed changing the septa prior to calibrating. Updated sequence to include running the EVAL. Renumbered the sequence. Updated name from AR32X to AR323
Calibration 2.a	Reflects current standard code	Updated name from AR32X to AR323 and renumbered section.
Calibration 5.e.	Reflects current process Old references not needed	Added North Carolina criteria for linear fit. Removed old reference numbers SOP-PP-031, SOP-PP-013, and SOP-PP-032
Procedure 2.a	Old reference not needed	Removed old reference number SOP-PP-011
Procedure 4	Reflects current practice	Added hexane as the solvent used for dilutions
Calculation	Old reference not needed	Removed old reference number SOP-PP-040
Statistical Information/Method Performance	Reference not needed	Removed reference 1-P-QM-QMA-9017309
Quality Assurance/Quality Control	Clarification Old reference not needed	Moved QC acceptance limits statement from Statistical Information/Method Performance section to this section. Removed old reference number SOP-PP-002
Appendix I C.	Reflects current sequence Reflects current MS and SS spikes	Added EVAL and renumbered sequence Updated names of parent solution and solvent used for each spike preparation

Revision: 7	**Effective Date:**	Jul 21, 2015
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version

Revision: 7		**Effective Date:** Jul 21, 2015
Historical/Local Document Number	Current relevant LIMS analysis scan for procedure	Added analysis scan 13713
Scope	Reflects current analysis scans and reporting limits	Updated LOQ for Tissue to 34 ug/kg and added tissue microwave scan 13713
Calibration	Enhancement	Added information on analyzing the DDT/endrin breakdown for retention time information.

Reference

1. *Test Methods for Evaluating Solid Waste*, SW-846, Method 8082A, February 2007.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0819, 11128, 11132, 11135	Ultrasonic Extraction for PCBs in a Solid Matrix by Method 3550C
Analysis #10497, 11140, 13100	Microwave Extraction Method 3546 for PCBs in a Solid Matrix
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015493	QC Data Acceptability and Corrective Action
1-P-QM-PRO-9015494	Interpretation of Chromatographic Data
1-P-QM-PRO-9015495	Preventative and Corrective GC Maintenance
1-P-QM-PRO-9015496	Monitoring QC Data Acceptance Limits
1-P-QM-PRO-9015498	Setting Up Single Component Initial Calibrations
1-P-QM-PRO-9015499	Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
1-P-QM-PRO-9015501	Common Equations Used During Chromatographic Analyses

Scope

This method is used for identifying and quantitating the following PCBs in solid samples and wipes using SW846 8082A:

Compound	Soil LOQ (µg/kg)	Wipes LOQ (µg)	Tissue LOQ (µg/kg)
Aroclor 1016	17	0.5	34
Aroclor 1221	17	0.5	34
Aroclor 1232	17	0.5	34
Aroclor 1242	17	0.5	34
Aroclor 1248	17	0.5	34
Aroclor 1254	17	0.5	34

Aroclor 1260	17	0.5	34
Aroclor 1262	17	0.5	34
Aroclor 1268	17	0.5	34

Aroclors 5422, 5432, and 5460 can be analyzed upon request of the client. See Appendix I for GC operating conditions and calibration information.

Compound	Soil LOQ (µg/kg)
Aroclor 5422	33
Aroclor 5432	33
Aroclor 5460	33

Limits of Quantitiation (LOQs) are based on statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

Analysis LIMS scan	Extraction LIMS scan	Description (targets)
10592	11132 sonication	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262, 1268
10885	10497 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262, 1268
13099	13100 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, 1268 Aroclor 5432, 5442, 5460
12718 wipes	10497 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262,1268
13219 tissue	11128 sonication	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262,1268
13713 tissue	10497 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262,1268

Basic Principles

A solid sample or entire wipe is extracted using sonic probe with 1:1 methylene chloride:acetone, or microwave extraction with hexane. The extract is dried, concentrated, and exchanged to hexane. A florisil and sulfuric acid cleanup is utilized to reduce any matrix interferences. The PCBs are then identified and quantitated using gas chromatography (GC) with electron capture detector (ECD). Copper cleanup may also be employed to reduce elemental sulfur or other matrix interferences which introduce large, unresolvable peaks into the chromatogram. Refer to 1-P-QM-PRO-9015477 for details on each cleanup procedure.

Solid matrices other than soil can also be analyzed as long as the sample can be handled through the extraction technique. Typically solids are reduced to small pieces for extracting (concrete, wood, other plant material etc). Tissue samples are ground and homogenized prior to extracting. Tissue samples may be whole fish, filets, or other miscellaneous species (usually aquatic but not necessarily). Wipe samples are analyzed by taking the entire wipe sample and any solvent in the sample container.

Reference Modifications

Gas Chromatography conditions are different than those listed in Method 8082A however, all QC criteria are met.

Interferences

Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

An electron capture detector (ECD) is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.

Extracts may require further cleanup by sulfuric acid, florisil, or copper if interferents such as oxygenated organics, unsaturated organics, and elemental sulfur are present. Refer to 1-P-QM-PRO-9015477 for details on each cleanup procedure.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Safety glasses must be worn around the GC where solvents and sample extracts are handled.

GC vials are disposed of in the designated lab container and subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the laboratory then taken to the lab-wide facility by personnel trained in hazardous waste disposal.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass containers with Teflon™-lined caps and kept cool at 0° to 6° C, not frozen. Sample extraction must be performed within 14 days of collection, and sample analysis must be performed with 40 days of extraction. Extracts are stored in the freezer at ≤-10°C.

Apparatus and Equipment

1. HP 7890 gas chromatograph equipped with an electron capture detector or equivalent

2. Columns:

   a. Phenomonex MultiRes I – 30 m × 0.32 mm × 0.5 µm or equivalent

   b. Phenomonex MultiRes II – 30 m × 0.32 mm × .25 µm or equivalent

   c. Alternatively, Restek columns can be used.

3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.

4. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents

1. Hexane for autosampler rinse vials. Stored at room temperature.

2. UPC (Ultra pure carrier) helium for carrier gas

3. UPC nitrogen for detector make-up gas

4. UPC hydrogen for carrier, either bottled or from a generator

B. Standards

1. All standards are prepared using Class A volumetric pipettes, syringes, and flasks.

2. An analytical balance is used to measure all weights.

3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

4. All prepared standard solutions are stored at ≤-10°C in labeled containers or vessels.

5. Aroclor 1016 & 1260 stock – Restek #32039 at 1,000,000 ppb in isooctane or equivalent.

a. At least two different lots of this material are kept in supply; one for working calibration standards and one for matrix spike.

b. Prepare a ten−fold dilution for an intermediate.

6. Aroclor 1016 ICV Stock – Accustandard Cat# C-2165-H-10X at 1,000,000 ppb in Hexane.

7. Aroclor 1260 ICV Stock - Accustandard Cat# C-2605-H-10X at 1,000,000 ppb in Hexane.

8. Aroclor 1221 stock – Restek #32007 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

9. Aroclor 1232 stock – Restek #32008 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

10. Aroclor 1242 stock – Restek #32009 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

11. Aroclor 1248 stock – Restek #362010 at 1,000,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source

Prepare an intermediate by diluting 1 mL to 10 mL of hexane. .

12. Aroclor 1254 stock – Restek #32011 at 1,000,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

13. Surrogate stock (SS) – Supelco #861284 containing DCB/TCX at 200,000 ppb in acetone or equivalent.

Prepare an intermediate by diluting 1.0 mL to 25 mL of hexane for AR1016/1260, AR1254, AR1248.

14. 1016/1260 Matrix spike (MS) stock – Restek #32039 at 1,000,000 ppb in hexane or equivalent.

15. Aroclor 1262 stock – Restek #32409 at 1,000,000 ppb in hexane or equivalent.

Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

16. Aroclor 1268 stock – Restek #32410 at 1,000,000 ppb in hexane or equivalent.

Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

17. Instrument Blank (IBLK) – Surrogate Stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 2000,000 ppb in acetone or equivalent.

18. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

d. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
PCB Spike	Aroclor 1016/1260 MS Stock	1.25	250	Acetone	PCB Spike
SS	Pest Surr Stock	1.5	1000	methanol	SW-846 Water surrogate – identical to that prepared for Pest/PCB analyses

19. An initial calibration verification standard must also be prepared at a concentration at the mid-point of the calibration for 1016, 1260,1248 and1254 using a stock solution purchased from a different source other than that used for the calibration standards.

20. Additional standards and preparations are listed in Appendix I for Aroclor 5442, 5432, and 5460 (PCT analysis).

Extraction

See Organic Extraction Analysis #0819, 11128, 11132, 11135 or Analysis #10497, 11140, 13100

Gas Chromatographic Conditions

The conditions listed are usually the optimum operating conditions but can vary to improve the sensitivity, linearity, and overall chromatography or shorten run times on each GC system.

Detector	ECD
Detector Temperature	330°C
Oven Temperature	110°C 40°C/min to 250°C 20°C/min to 280°C 30°C/min. to 330°C Hold until DCB elutes ~ 2 min.
Carrier	Hydrogen at 12 psi, 5ml/min constant flow (Can be substituted with Helium)

Makeup gas	N2 at 30 mL/min. for Varian GCs N2 at 55 mL/min. for HP GCs
Injection size	1 µL, direct injection
Injection Temperature	225°C

A Merlin microseal may be used in place of a traditional septum.

Calibration

1. Fill the autosampler rinse vials with clean solvent or replace vials which appear dirty.

2. Prepare a sequence using the following suggested order of injections:
    1. Conditioner
    2. IBLK
    3. EVAL
    4. AR161
    5. AR162
    6. AR163
    7. AR164
    8. AR165
    9. AR481
    10. AR482
    11. AR483
    12. AR484
    13. AR485
    14. AR541
    15. AR542
    16. AR543
    17. AR544
    18. AR545
    19. AR213
    20. AR323
    21. AR423
    22. AR623
    23. AR683
    24 AR16xx
    25. MD16 (MDL)
    26. IC16x (ICV)
    27. IC48x (ICV)
    28. IC54x (ICV)
    29. Blank
    30. LCS
    31. 1234567
    32. 1234567ms
    33. 1234567msd
    34 – 44. Continue running samples
    45. AR163 (CCV)

46. IBLK

a. For routine PCBs:

(1) The AR21, AR32, and AR42 are level 3's in the cal and are entered at AR213, AR323, and AR423 in the cal.

(2) AR62 and AR68 are both level 3s and are listed as AR623 and AR683 in the sequences.

(3) A single point of Aroclor 1016 is analyzed with the calibration to aid in better pattern recognition in the samples. Aroclor 1016 and 1242 have similar patterns with the exception of the smaller trailing peaks at the end of the Aroclor 1242 pattern. This is identified in the sequence as AR16xx.

(4) A DDT/Endrin breakdown standard is run with each ICAL in order to provide the retention times for p,p-DDE, p,p-DDD, and p,p-DDT which may interfere with the pattern for aroclor 1254 and 1260.

b. See Appendix I for a sequence example when the analysis of PCTs (Aroclors 5442, 5432, and 5460) is requested.

3. Inject conditioner to prime the system.

a. The conditioner injection is usually a standard or sample which has already been injected.

b. It is used to prime the system and is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

c. Hexane blanks may also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

4. An instrument blank (IBLK) is always run prior to a new calibration to confirm that the instrument is free of background noise or contamination.

IBLK may also be run with the continuing check standards - this is optional but frequently requested for projects.

5. Initial Calibration (ICAL)

a. Calibrate for the aroclors using the five levels of 1016, 1260, 1248, and 1254 (calibration range of 50 µg/L through 1000 µg/L) and using the single point for 1221, 1232, 1242, and 1262 and 1268 when needed (200 µg/L standard used).

b. As an option, 1248 and 1254 may use a single point calibration.

c. An external standard calibration based on the average calibration factor (AVG CF) is used for quantitation where the %RSD is ≤20%.

d. The surrogate standards are calibrated using the AR16 levels and the calibration is also performed using AVG CF unless the %RSD is >20%.

e. When the % RSD criteria is not met (i.e.> 20%), a linear calibration curve is used.

(1) The curve must meet a correlation coefficient of 0.99 to be a valid fit.

(2) Extrapolate or force to zero is not allowed. Set the zero to ignore.

(3) See 1-P-QM-PRO-9015498 for more details.

(4) North Carolina requires calculation of relative standard error (RSE) for any linear fits. The criteria is <50%

RSE = ((true – calc)/true) x 100

f. If the %RSD criteria or 0.99 curve coefficient cannot be met:

(1) Inspect the data points to see if one or more calibration levels may have concentrated due to solvent evaporation or degraded over time.

(2) Reinject or remake the standard if this is the cause.

(3) Perform instrument maintenance as needed.

(4) See 1-P-QM-PRO-9015495 for troubleshooting linearity problems.

A quadratic fit may not be used for South Carolina samples.

g. Set up the aroclor calibration data in a custom program under Datalog.

(1) The retention times of the peaks to use for identifying and quantifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations (in µg/L).

(2) See 1-P-QM-PRO-9015499 for details.

h. Ensure the surrogate peaks in the standards are labeled properly.

i. Set the scaling of chromatograms so that the size of the peaks of interest for each aroclor are approximately 2 to 3 mm in height at the concentration of the method detection limit (MDL).

(1) Ensure all peaks in the MDL standard are integrated.

(2) By running the1016/1260 MDL standard, the majority of peaks for all aroclors should be represented.

6. Initial Calibration Verification (ICV)

a. Verify the calibration curves using the ICV mixtures injected directly after the full ICAL.

b. The % difference of the concentrations for these must be within 20% difference of the nominal concentration.

7. Continuing Calibration Verification (CCV)

a. Calibration verification is performed after each set of twenty injections (samples, QC, blanks, etc.) or 12 hours, whichever comes first.

b. Use AR16 to evaluate the calibration of the aroclors (or other aroclors as requested for particular clients or projects).

c. The concentration quantitated for the continuing calibration verification standards must be within 20% difference (%D) of the nominal concentration for each compound.

d. Samples must be bracketed by compliant standards.

(1) Exception: If standard following a sample is outside the ±20% but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected.

(2) If CCVs continue to fail, corrective action must be taken, which can include performing injection port maintenance.

e. If confirmation of target analytes is needed, then the second column should meet the 20% continuing calibration criteria, as well as all initial calibration criteria.

8. The instrument blank (IBLK) is injected after each set of continuing calibration verification when requested.

a. It must be evaluated as a water matrix against the water MDL/LOQs.

b. The IBLK must not have any target compounds above the reporting limits.

(1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be injected after another compliant IBLK.

(3) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up the instrument.

9. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours, or at no less than ±0.02 min, applied to the midpoint initial calibration standard.

b. If the RTs for a continuing calibration verification standard fall outside the RT window, update the midpoint RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24−hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Prepare a sequence of injections as suggested in the Calibration Section, along with the appropriate check standards.

2. Retention times of peaks in the samples are compared to the standard RT windows

a. Peaks present on both columns that are also in the correct ratios to represent an aroclor are quantitated, and the high value is reported unless chromatographic anomalies are observed. See 1−P−QM−PRO−9015494.

b. Use a minimum of three to six peaks for quantitation, with the exception of certain mixes where it may be more accurate to use less peaks to avoid excessive overlap of patterns.

3. Continue running groups of samples/injections followed by check standards every 12 hours or 20 injections, whichever comes first.

a. For projects where a known aroclor is present and at the request of clients, other aroclors can be run for the continuing check standard. For instance, a set of continuing standards may be AR483, AR163, IBLK.

b. Aroclor 1262 and 1268 can be analyzed when requested. A full five point curve can also be run, depending on the project requirements.

4. If significant interference is present, schedule florisil and/or sulfuric acid cleanup. If elemental sulfur is present, TBA or copper the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract with hexane and adjust LOQs accordingly.

5. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

1. The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each aroclor.

Usually, the six major peaks that are unique to each aroclor are chosen for quantitation, with the exception of 1221 where only three peaks are available.

2. Sample concentrations are calculated per peak using average calibration factor (AVG CF) from the initial calibration.

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{FV}{IW} \times DF = \mu g/kg \text{ as received per peak}$$

Where:

FV = Final volume (mL)

IW = Initial weight (g)

DF = Dilution factor, as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(\text{Result 1} + \text{Result 2} + \ldots + \text{Result n}) / n = \text{Average Result}$$

3. The surrogate results are determined using either AVGRF or linear curve:

Using AVGCF from the initial calibration:

$$\frac{Sample\ Height}{AVCRF\ (CF)} \times \frac{FV}{IW} \times DF = \mu g/kg \quad as\ received$$

b. Using linear curve from the initial calibration:

$$[(Sample\ Height - Y - intercept)/Slope] \times \frac{FV}{IW} \times DF = \mu g/kg\ as\ received$$

Where:

FV = Final volume

IW = Initial weight

DF = Dilution factor, as needed

4. See 1-P-QM-PRO-9015501 for all other equations related to the analysis.

Statistical Information/Method Performance

Initially, perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire extraction and analysis procedure. The results are tabulated using an Excel spreadsheet. Compare and pool results to determine the final reporting MDL. An MDL study or verification of the MDL is required each year. NELAC allows for an annual verification of the MDL in lieu of a full MDL study. The department supervisor maintains annual study data. Updates to the LIMS are made as need by the QA Department and only as directed by the manager. Update the department database via a download from the LIMS.

Common single component pesicides such as DDT, DDD, and DDE may cause interference with the aroclor pattern. To ensure that the analyst is aware of this, a standard containing DDE/DDD/DDT will be run with each initial calibration.

Qaulity Assurance/Quality Control

QC Acceptance limits are established as statistical limits. See 1-P-QM-PRO-9015496 further information on monitoring and establishing limits.

A sodium sulfate blank and a sodium sulfate spike (LCS) are analyzed with every group of samples up to a maximum of 20. An MS/MSD is performed per batch. For wipes and when an MS/MSD cannot be performed, an LCSD will be extracted.

Aroclor 1016 and 1260 are routinely spiked; however, other aroclors can be spiked as requested by clients.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

See 1-P-QM-PRO-9015493 for details on QC acceptance criteria and corrective action.

Appendix I

PCT Analysis (Aroclor 5422, 5432, and 5460)

A. Standards:

1. PCT-Stocks- Aroclor 5432, 5442, and 5460 purchased as individually ampulated solutions from Accustandard at 35,000ug/L.

2. Surrogate Stock- Ultra ISM-320 containing TCX/DCB at 200,000ppb in acetone. Prepare an intermediate by diluting 0.25mL to 25mL of hexane.

3. MS stock of Aroclor 5442 - Absolute cat# 71791, 1000ug/L in Methanol.

4. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

d. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
MS	Aroclor 5422 Stock	0.75	100	Acetone	Aroclor 5442 PCT Spike
SS	Pest Surr mix	1.5	1000	methanol	SW 846 surrogate

B. GC Chromatographic Analysis

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make any changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity, and/or improve separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

Detector: ECD

Detector temp: 330°C

Oven Temp: 110°C to 250° at 40°C/min, to 280°C at 20°C/min, to 330°C at 30°C/min, hold 9 min.

Carrier: Hydrogen at 3.6 mL/min

Makeup gas: N_2 at 60mL/min or equivalent

Injection size: 1-uL, direct injection

Injection temp: 225°C

C. Example of a Sequence:

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR481
10. AR482
11. AR483

12. AR484
13. AR485
14. AR541
15. AR542
16. AR543
17. AR544
18. AR545
19. PCT1
20. PCT2
21. PCT3
22. PCT4
23. PCT5
24. A4421
25. A4422
26. A4423
27. A4424
28. A4425
29. AR213
30. AR323
31. AR423
32. AR623
33. AR683
34. AR16xx
35. MDPCTX
36. MD16
37. IC16
38. IC48
39. IC54
40. Blank
41. LCS
42. 1234567
43. 1234567MS
44. 1234567MSD

45-59. Continue running samples

60. AR163
61. PCT3
62. A4423
63. IBLK

End of document

Version history

Version	Approval	Revision information
8	26.AUG.2016	

eurofins	Always check on-line for validity **Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10920**		
Old Reference: **1-P-QM-WI -9015079**		
Version: **19**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **07-OCT-2016**	Document users: **5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Pest/PCB Waters, 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis DOD - 6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187

Revision Log

Revision: 19		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects current analysis numbers	Added Analysis #13634, 14134, 14166, 14169, 14184, 14186, 14188
Apparatus and Equipment	Reflects current practice	Added Automated Water Extraction Bench, Rapid-Vap Evaporator, and Rapid-Vap Evaporator Tube

Revision: 19		**Effective Date:** This version
Procedure 6.b. & 6.c.	Reflects current practice	Added surrogate and spike details for new analysis numbers
Procedure 11-19	Reflects current practice	Separate 1L extraction from 250 mL extraction and added new procedure for 250 mL extraction

Revision: 18		**Effective Date:** Apr 13, 2016
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Apparatus and Equipment	Reflects current process	Added Turbo Vap tubes and Turbo Vap
Procedure 13	Reflects current process	Added 13.b for analyses that require Turbo Vap concentration
Procedure 18	Reflects current process	Added 18.b for analyses that require Turbo Vap concentration
Procedure 19	Reflects current process	Added 19.b, 19.c, and19.d to differentiate between which steps require Turbo Vap concentration and which steps require steam bath concentration.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3510C, Rev. 3, December 1996.
2. USEPA, 40 CFR Part 136, Appendix A, Method 608.
3. USEPA, 40 CFR Part 136, Appendix A, Method 622.
4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0177, 0950, 0180, 1954	Pesticides in Water by Method 8081A using GC-ECD
Analysis #2257, 2253	Captan and Captafol by Method 8081A in Waters and Solids using GC-ECD
Analysis #5366, 10410, 10593, 12144, 13182, 13186	Organophosphorous Pesticides by Methods 8141A/8141B/622 in Aqueous Samples using GC-NPD
Analysis #6030, 10227	Polychlorinated Biphenyls (PCBs) by Method 608 or 8082 in Waters
Analysis #7572	Pesticides in Aqueous Samples by Method 608
Analysis #10589, 10647	Pesticides in Water by Method 8081B using GC-ECD
Analysis #10591, 13092	Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples using GC-ECD
	Low Level PCBs in Water by Method 8082/8082A using GC-ECD

Analysis #12013, 12686	
1-P-QM-PRO-9015407	Pesticide Extract Cleanup Using Gel Permeation Chromatography
1-P-QM-PRO-9015475	Glassware Cleaning for Organic Extractions
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Purpose
The purpose of this SOP is to provide clear instructions for performing the separatory funnel extraction procedure on samples that are to be analyzed for pesticides and PCBs.

Scope
This procedure is for the extraction of organochlorine and organophosphorous pesticides and PCBs from wastewaters.

Basic Principles
An aliquot of the sample is placed into a separatory funnel. The volume of sample extracted is adjusted (if appropriate) depending on the physical appearance of the sample and the volume sent for analysis. A surrogate standard is added to the sample to monitor recovery. The sample is then extracted with methylene chloride. The extract is dried, concentrated, and exchanged to hexane. Several cleanup procedures are available to eliminate matrix interferences before the sample is analyzed. They include sulfuric acid treatment, copper treatment, florisil, and Gel-Permeation Cleanup (GPC).

Reference Modifications
1. Surrogate and matrix spiking solutions are not added before the transfer to the extractor. For several reasons:

a. Samples must be poured from the amber bottles to determine the matrix and volume of sample to use for each extraction.

b. Many sample bottles have no headspace and there is no room to add surrogate to the sample in the bottle.

c. Due to the volume of samples extracted, a separate graduated cylinder for each sample is unrealistic.

d. To maintain consistency with all extractions, no samples are spiked in the bottle or graduated cylinders.

2. In Procedure Step 19 the joint of the KD is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

Interferences

Impurities in solvents, reagents, glassware, or other hardware used in sample processing interfere with the method. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the employees training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the extraction and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, or one blind sample.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass bottles with PTFE-lined lids, preserved with sodium thiosulfate, and stored refrigerated at 0 - 6°C, not frozen. Sample extraction must be started within 7 days of collection. Extracts are stored frozen at ≤-10°C.

Apparatus and Equipment

1. Separatory funnel for extracting organic components from an aqueous matrix

2. Kuderna-Danish (K-D) assembly with appropriate ampule for extracting the solvent used during the extraction

3. Water bath – VWR/LLI Model #1127 or equivalent

4. Sodium sulfate column with extra course frit

5. Nitrogen evaporation (N-Evap) with nitrogen supply – Organomation Associates or equivalent

6. pH paper – Wide range

7. Automatic shaker – Glass Col or equivalent, capable of holding 2-L separatory funnels

8. Pipettes – Class A, assorted sizes

9. Graduated cylinders – Class A, assorted sizes

10. Pipettes – Disposable

11. Solvent dispenser – Brinkmann, adjustable or equivalent

12. Balance – Capable of weighing to 0.01 g

13. Centrifuge – Beckman GS-6 or equivalent

14. Micro-Snyder columns

15. Wash bottles – Teflon

16. Vials – Assorted sizes

17. Teflon boiling chips

18. Syringes - Assorted sizes

19. Micro-pipetter

20. Turbo Vap – Zymark Turbo Vap II concentration station or equivalent

21. Turbo Vap tubes- 250ml Zymark tubes or equivalent

22. Automated Water Extraction Bench – capable of holding separatory funnels for 250 mL extractions.

23. Rapid-Vap Evaporators – for concentration of 250 mL extractions

24. Rapid-Vap Evaporator Tubes – Class A

Reagents and Standards

1. Methylene chloride (CH_2Cl_2) – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

2. Acetone – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

3. Hexane – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

4. 10N Sodium hydroxide (NaOH) – Lab Chem or equivalent. Store at room temperature. Follow manufacturer's expiration date.

5. Sulfuric acid (H_2SO_4) – ACS grade or equivalent. Store at room temperature. Follow manufacturer's expiration date.

6. Sodium Sulfate (Na_2SO_4) – Reagent grade or equivalent. Bake at approximately 400°C for a minimum of 4 hours in a shallow pan to remove organic contaminants. Store in a glass jar at room temperature for up to 1 year after baking.

7. Reagent water – water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the reagent water supplied at the taps in the laboratory meets this criterion. If the reagent water does not meet the requirements, see your supervisor for further instructions.

8. Extraction fluid – Prepared and delivered by the leachate department. Store refrigerated at 0-6^{o}C, not frozen, in a glass container with a PTFE-lined lid.

9. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical
Analysis #0177, 0950, 0180, 1954,14134; Analysis #2257, 2253; Analysis #5366 10410, 10593, 12144, 13182, 13186; Analysis #6030, 10227, 14169, 14188; Analysis #7572, 13634; Analysis #10589, 10647, 14166; analysis 10591, 13092, 14184, 14186 ; and Analysis #12013, 12686.

Calibration

Not applicable to this procedure.

Preparation of Glassware

See SOP 1-P-QM-PRO-9015475.

Procedure

1. Determine the volume of sample to be used for each extraction. Typically, this is 1L for routine extractions or 250mL for 250mL extractions.

1 Liter Analyses: 0177, 0180, 1954, 2257, 2253, 5366, 10410, 10593, 12144, 13182, 13186, 6030, 10227, 7572, 10589, 10647, 10591, 13092, 12013, 12686

250 mL Analyses: 13634, 14134, 14166, 14169, 14184, 14186, 14188

a. If uncertain of the volume to extract for any sample, ask your supervisor.

b. Use one full bottle for all analysis scans (with the exception of scan #0950) unless the matrix is poor (thick, lots of sediment, extremely foul odor).

(1) If using reduced volume due to matrix, take as much as possible while trying to minimize matrix problems, document why the reduced volume was used.

(2) Reduced volume aliquots due to matrix are typically are 500, 200, or 100 mL.

c. For analysis 0950:

(1) If the sample bottle contains at least 500 mL of sample, measure 200 mL.

(2) If the sample bottle contains <500 mL of sample, use 1/2 of the available volume or 10 mL, whichever is greater.

(3) If a matrix spike (MS) or MS/matrix spike duplicate (MSD) is required for the sample, use 200 mL each.

d. The background, MS, and MSD are performed on three separate aliquots of a field sample.

2. Prepare the blank, laboratory control sample (LCS), laboratory control sample duplicate (LCSD) (if applicable) with 1 L of reagent water (or 250mL for 250 mL extractions) measured into the separatory funnel.

Exception: For Analysis #0950 - The blank, LCS, and LCSD (if applicable) are prepared using 200 mL of extraction fluid measured into the separatory funnel.

3. For samples using 1 entire bottle:

a. Etch the outside of the bottle with a scriber at the meniscus.

b. Shake the bottle vigorously and then pour the contents into a separatory funnel.

4. For all samples requiring a specified volume:

a. Shake each bottle vigorously.

b. Use a clean graduated cylinder to measure the desired volume.

c. Use a wash bottle to rinse the graduated cylinder with methylene chloride and add the rinsate to the separatory funnel.

d. If <1000 mL (or <250mL for 250mL extractions) of sample is used, use a graduated cylinder to add enough reagent water to bring the volume in the extractor to 1−L (or 250mL for 250mL extractions).

5. Record any comments about the sample and the initial volume, if known at this time, on the extraction sheet.

6. Use pipettes to add surrogate standards and spiking solutions to the aqueous sample in the separatory funnel.

a. Be certain the standard drips directly into the aqueous sample without touching the glass side of the separatory funnel to avoid poor recoveries.

b. Surrogates – Surrogates are added to all samples, blanks, and spikes.

The type of surrogate is determined by the analysis scan number. Typically they are as follows:

(1) For analyses 0177, 0180, 0950, 1954, 2257, 6030, 7572, 10589, 10591, 13092, 10647, 13092 – 1.0 mL SW−846 Surrogate.

(2) For analysis 5366, 10593, 10410, 12144, 13182, 13186 – 1.0 mL NP Surrogate

(3) For Analysis #12013, 12686 – 0.1 mL of SW-846 Surrogate

(4) For Analysis #10227 – If entered for prep 11117 use 1.0 mL of SW846 Surrogate. If entered for prep 13086 use 1.0 mL of 2mL SW846 Surrogate.

(5) For Analyses #13634, 14134, 14166, 14169, 14184, 14186, 14188 – Use 1.0 mL of Mini SW846 Surrogate

c. Spiking Solutions – Spiking solutions are added to the LCS, LCSD (if applicable), MS, and MSD.

(1) The type of spike is determined by an analysis number. Typically they are as follows:

(a) For analysis 6030, 10591, 13092 – 1.0 mL PCB Spike

(b) For analysis 10227 – If entered with prep 11117 use 1.0 mL of PCB spike. If entered with prep 13086 use 1.0 mL of 2mL PCB spike.

(c) For analysis 7572, 10589, 0177 – 1.0 mL SW-846 Spike

(d) For analysis 0950 & 10647 – 1.0 mL TCLP Pesticides Spike regardless of initial volume

(e) For analysis 5366 – 1.0 mL Triazine Herb Spike

(f) For analyses 10593 and 10410 – 1.0 mL OPPA Spike

(g) For analysis 2257 – 1.0 mL Captan/Captafol Spike

(h) For analysis 0180 – 1.0 mL of Chlordane spike

(i) For analysis 1954 – 1.0 mL of SW846 spike and an additional LCS/LCSD with 1.0 mL of Alaclor

(j) For analysis 12013 – 0.1 mL of PCB Spike

(k) For Analysis 13093 – 1.0 mL of PCB Spike and prepare a separate LCS/LCSD using 1.0 mL of 5442 Arochlor Spike

(l) For Analysis 13182 and 13186 – 1.0mL of Appendix IX Water Spike (or 0.25 mL of Appendix IX water Spike for 250mL extractions).

(m) For analysis 13634, 14134, 14166 – 0.25 mL of SW846 Spike

(n) For analysis 14169, 14184, 14188 – 1.0 mL of Mini PCB Spike

(o) For analysis 14186 – 1.0 mL of Mini PCB Spike and prepare a separate LCS/LCSD using 1.0 mL of Mini 5442 Arochlor Spike

(2) Spike details can be found in the corresponding analytical SOP(s)

(3) This is changed to accommodate specific-client requirements, if appropriate.

(4) If a sample requires any special compounds in addition to the standard list, an appropriate spike containing those compounds is added at this time.

(5) See SOP 1-P-QM-PRO-9015490 for storage and handling of spikes.

7. Measure and record the pH of the sample using wide-range pH paper.

a. If necessary, adjust the pH to between 5 and 9 using 10N NaOH (to bring up the pH) or concentrated H_2SO_4 (to lower the pH).

b. To adjust the pH, add a few drops of the appropriate solution with a disposable pipette, shake the separatory funnel and re-check the pH.

c. If the pH now falls between 5 and 9, record the adjusted pH on the extraction sheet.

d. If the pH is still out of range, continue adding small aliquots of base or acid, shaking and checking the pH until it is within the specified range.

e. Record the adjusted pH.

NOTE: Any samples from North Carolina require that the volume of acid or base added to the sample to adjust the pH to be recorded on the extraction sheet.

8. If the original sample bottle is empty:

a. Use a solvent pump to measure 60 mL (15 mL for 250 mL extractions) of methylene chloride. Add the methylene chloride to the sample bottle. Then cap the bottle and invert several times. Add the solvent to the separatory funnel.

b. Fill the bottle to the marked level with water and transfer the water to a graduated cylinder to determine the initial volume.

c. Alternatively, for all analyses **except Analysis #7572, and analysis 6030 (Method 608)** weigh the empty bottle and tare the balance.

(1) Fill the bottle to the marked level with water and place the bottle onto the tared balance.

(2) This weight rounded to a whole number is the initial sample volume.

d. Record the initial volume on the extraction sheet.

9. If the sample container is not empty, measure 60 mL (15 mL for 250mL extractions) of methylene chloride and add the solvent directly to the separatory funnel.

10. Cap the funnel, invert it, and vent immediately.

11. For 1 Liter Analyses: Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

For 250 mL Analyses: Separatory funnels remain on the Automated Water Extraction Bench. Press the green “start” button to lower the hood and activate the Automated Water Extraction Bench. The bench will tumble for 2 minutes. The sash will rise after the samples have tumbled.

NOTE: Shaker speeds vary greatly between instruments so the proper setting is marked on each.

12. For 1 Liter Analyses: Place the separatory funnel on the rack and allow it to sit undisturbed for approximately 10 minutes. For 250 mL Analyses: the separatory funnel will sit undisturbed on the Automated Water Extraction Bench.

a. The time required for extracts to set undisturbed is based upon visual confirmation that the layers are adequately separated. Additional time may be necessary for samples with unusually high density (i.e. high salt content).

b. If an emulsion forms and is greater than 1/3 of the volume of the solvent layer, mechanical techniques such as stirring and centrifugation must be employed to complete the separation.

13 Removing the Solvent Layer.

a. For 1 Liter analyses (except 5366, 10593, 10410, 12144, 13182, and 13186) : Remove the solvent layer by opening the stopcock and collect the solvent in a metal beaker then pour through approximately 10cm of sodium sulfate into a K-D apparatus containing a Teflon boiling chip.

b. For analyses 5366, 10593, 10410, 12144, 13182, and 13186, remove the solvent layer by opening the stopcock and collecting the solvent in a metal beaker. The solvent layer is then poured into a sodium sulfate column and collected in a Turbo Vap tube.

c. For 250 mL analyses: drain the solvent layer directly into the sodium sulfate column containing approximately 5cm of sodium sulfate. This drains into a Rapid-Vap Evaporator Tube.

NOTE: Do not use more than the recommended quantity of sodium sulfate and take care not to transfer water into the column. Excessive sodium sulfate and/or water in the column results in low aldrin recovery.

14. Use a solvent pump to add 60 mL (15mL for 250mL extractions) of methylene chloride to the separatory funnel and repeat Procedure Steps 10 through 13, venting only as necessary.

15. Again, use a solvent pump to add 60 mL (15 mL for 250mL extractions) of methylene chloride to the separatory funnel and repeat Procedure Steps 10 through 13, venting only as necessary.

16. For 1L extractions only, rinse the metal beaker that was used for solvent collection with approximately 20 mL of methylene chloride. Pour into salt column.

17. Rinse salt column with approximately 20 mL (5mL for 250mL extractions) of methylene chloride. Use a hand held bulb to squeeze through any excess methylene chloride.

18. Concentrating the Extract.

a. For 1 Liter Analyses: Attach a 3−ball Snyder column to the K−D, wet with solvent, and concentrate the extract to approximately 3 to 5 mL on a steam bath at 85° to 99°C. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. This steam bath temperature ensures concentration in a reasonable length of time.

b. For 250 mL Analyses: Place the Evaporator Tube containing the solvent extract into the pre-heated (approximately 80°C) Rapid-Vap Evaporator. Set the timer for 15 minutes and speed at 75% (the speed is the rotation within the unit while the sample is evaporating). Confirm that the nitrogen is set at 15 psi (this is adjusted on the regulator attached to the nitrogen line). Allow samples to concentrate to between 1-2mL.

Note: The temperature, timer, and speed settings can be adjusted as necessary to accommodate difficult sample matrices, etc.

c. For analyses 5366, 10593, 10410, 12144, 13182, and 13186, place the Turbo Vap tubes in a Turbo Vap with the temperature set between 45°and 50°C.

To avoid loss of analytes do not allow the ampule or Turbo Vap tube to go dry

19. Allow the sample to cool for 10 minutes.

a. If the sample does not require GPC and a steam bath concentration or Rapid – Vap evaporation is required:

(1) Use a graduated cylinder to add approximately 50 mL of hexane directly to the K–D through the Snyder column for 1 Liter analyses or directly to the Rapid-Vap Evaporator Tube for 250 mL analyses.

(2) For 1 Liter Analyses: Concentrate until a volume of 3 to 5 mL is achieved. For 250 mL Analyses: Concentrate to around 1 mL.

(3) Remove the sample from the bath (or Rapid-Vap Evaporator) and allow the sample to cool for 10 minutes.

(4) For 1 Liter Analyses: Remove the ampule and use a wash bottle to adjust the final volume to exactly 10.0 mL with hexane in a calibrated ampule. For 250 mL Analyses: Bring to final volume 2mL in the Class A Evaporator Tube by rinsing with hexane.

Exception:

(a) If samples are scheduled for Prep 12026, remove the ampule and place on an N-evap until the volume is below 5 mL. Adjust the final volume to 5 mL with hexane in a calibrated ampule.

(b) If samples are scheduled for Prep 13093,, remove the ampule and place on an N-evap until the volume is below 2mL. Adjust the final volume to 2mL with Hexane in a calibrated ampule.

(5) Mix extract thoroughly with a disposable pipette.

b. if the sample does not require GPC and a Turbo Vap concentration is required.

(1) Use a graduated cylinder to add approximately 50ml of hexane directly to the Turbo Vap tube.

(2) Concentrate until a volume of <2 ml is achieved.

(3) Remove the sample from the Turbo Vap. Quantitatively transfer the extract with hexane into a calibrated ampule and bring to a final of 10ml for 1liter preps and to 2ml for 250ml preps.

(4) Mix thoroughly with a disposable pipette.

c. If the sample requires GPC and a steam bath concentration or Rapid-Vap Evaporation is required.

(1) For 1 Liter Analyses: Remove the ampule and adjust the final volume to exactly 10.0 mL with methylene chloride. For 250 mL Analyses: Quantitatively transfer the sample from the Evaporator Tube and bring to FV 10.0 mL with methylene chloride using a Class A volumetric.

(2) Mix thoroughly with a disposable pipette.

(3) Perform GPC cleanup following SOP 1-P-QM-PRO-9015407.

(4) Once the GPC cleanup is completed, place the extract in a K-D containing a Teflon boiling chip.

(5) Attach a 3-ball Snyder column to the K-D, wet with solvent, and concentrate the extract to approximately 3 to 5 mL on a steam bath at 85° to 99°C. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

(6) Allow the sample to cool for 10 minutes.

(7) Use a graduated cylinder to add approximately 50 mL of hexane directly to the K-D through the Snyder column.

(8) Concentrate until a volume of 3 to 5 mL is achieved.

(9) Remove the sample from the bath and allow the sample to cool for 10 minutes.

(10) Remove the ampule and use a wash bottle to adjust the final volume to exactly 5.0 mL with hexane.

(11) Mix thoroughly with a disposable pipette.

d. If the sample requires GPC and Turbo Vap concentration is required.

(1) Remove the Turbo Vap tube from the Turbo Vap. Quantitatively transfer the extract into a calibrated ampule and bring to a final volume of 10ml with methylene chloride.

(2) Mix thoroughly with a disposable pipette.

(3) Perform GPC cleanup following SOP 1-P-QM-PRO-9015407.

(4) Once the GPC cleanup is completed, place the extract in a Turbo Vap tube.

(5) Place the Turbo Vap tube in a Turbo Vap with the temperature set between 45° to 50° C. Concentrate the extract to approximately 3 to 5ml.

(6) Use a graduated cylinder to add approximately 50ml of hexane directly into the Turbo Vap tube and concentrate to approximately 2ml.

(7) Remove the sample from the Turbo Vap. Quantitatively transfer the extract into a calibrated ampule using hexane to bring to a final volume of 5ml.

20. Complete Procedure Steps 21 through 24 as needed.

21. If the extract is scheduled for analysis 6030, 10227, 12013, 14169, 14184, 14186, 14188 treat the extract with sulfuric acid as described in SOP 1-P-QM-PRO-9015477.

22. If the extract is scheduled for analysis 0177, 7572, 10589, 13634, 14134, 14166 florisil the sample 2 mL to 2 mL as described in the Pesticide Florisil section of SOP 1-P-QM-PRO-9015477.

Pesticide florisil is also being performed for analysis 0950 and 2257 if required due to matrix or client request.

23. If the extract is scheduled for analysis 6030, 10227, 12013, 14169, 14184, 14186, 14188 florisil the extract following the PCB Florisil section of SOP 1-P-QM-PRO-9015477.

24. If the extract has a sulfur odor, perform copper cleanup of the extract as described in SOP 1-P-QM-PRO-9015477 for analysis 6030 only. Other samples are cleaned up with GPC.

Calculations

See analysis method as listed in the Cross Reference section.

Statistical Information/Method Performance

See analysis method as listed in the Cross Reference section.

Quality Assurance/Quality Control

A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. Exception: For analyses referencing Methods 608 or 622 (i.e. 7572, 6030, 11119, 11123,13634, 14188) batches cannot exceed 10 field samples.

For each batch of samples extracted a blank, an LCS, an MS and an MSD must be extracted. For method 608, the laboratory must, on an ongoing basis, spike at least 10% of the samples, if enough sample volume is received, to assess accuracy. If there is limited sample preventing the preparation of the MS/MSD, an LCSD must be prepared instead. If the batch contains only field or equipment blank samples, the LCS/LCSD QC pairing must be used.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed instead.

End of document

Version history

Version	Approval	Revision information
19	07.OCT.2016	

An error has happened on file: /D4Doc/formularer/2016/09/Quality Document # 1-P-QM-WI -9015079_18_665410241.doc
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eurofins	Always check on-line for validity **Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples using GC-ECD**	Level: **Work Instruction**
Document number: **T-PEST-WI9238**		
Old Reference: **1-P-QM-WI-9015109**		
Version: **5**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **04-NOV-2015**	Document users: **6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Pesticide Residue Analysis_Manager**

LIMS ID
Analysis DOD - 10591, 13092

Revision Log

Revision: 5		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Historical/ Local Document Number	LIMS scan relevant to this procedure	Add 13092 for PCTs

Revision: 5	**Effective Date:** This version	
Sample Collection, Preservation, and Handling	Reflects current industry standard for refrigeration storage.	Changed to 0° to 6° C, not frozen.
Apparatus and Equipment	Reflects current instrumentation	Updated 7890 GC
Calibration	Reflects method criteria Enhancement Reflects current procedure	Changed wording to second column should meet 20% continuing calibration criteria. Added information on analyzing an EVAL to aid in identifying possible DDD, DDE, and DDT in the aroclor pattern. Added information on analyzing a single PCB 1016 standard to help with pattern recognition. Changed the RT window from 0.03 to 0.02 min.
Gas Chromatographic Conditions	Reflects current operating conditions	Updated GC conditions.
Calculation	Clarification	Added information on not using individual peaks where the value is <MDL in calculating the PCB concentration.
Appendix I	Clarification for PCTs	Removed table for calibration standard preparation and added information referencing the standard database. Updated GC conditions for analyzing PCTs.

Revision: 4	**Effective Date:** Jan 10, 2013	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Cross Reference	Referenced in SOP	Added Demonstrations of Capability reference 1-P-QM-QMA-9015390
Scope	Clarification	Added PCT's (Aroclor 5422, 5432, and 5460).
Basic Principles	Unnecessary information	Removed 1L sample size.
Definitions	Enhancement	Added full definitions vs. acronyms.
Personnel Training and Qualification	Clarification	Added information on IDOC and DOCs.
Apparatus and Equipment	Enhancement	Added information on the data systems capability.
Reagents and Standards	Reflects current practices	Added reference to PCT's and to the standard database.
Extraction	Reflects current extractions	Deleted all unnecessary extractions scans.
Calibration	Enhancement	Reformatted section
Appendix I	Reflects current procedure	Added calibration information and sequence information.

Reference

1. *est Methods for Evaluating Solid Waste*, SW-846, Method 8082A, February 2007.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187	Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015493	QC Data Acceptability and Corrective Action
1-P-QM-PRO-9015494	Interpretation of Chromatographic Data
1-P-QM-PRO-9015495	Preventative and Corrective GC Maintenance
1-P-QM-PRO-9015496	Monitoring QC Data Acceptance Limits
1-P-QM-PRO-9015498	Setting Up Single Component Initial Calibrations
1-P-QM-PRO-9015499	Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
1-P-QM-PRO-9015501	Common Equations Used During Chromatographic Analyses
1-P-QM-QMA-9015390	Demonstrations of Capability
1-P-QM-QMA-9017309	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is useful for identifying and quantitating the following polychlorinated biphenyls (PCBs) as Aroclors in aqueous matrices:

Compound	LOQ (µg/L)
Aroclor 1016	0.5
Aroclor 1221	0.5
Aroclor 1232	0.5
Aroclor 1242	0.5
Aroclor 1248	0.5
Aroclor 1254	0.5
Aroclor 1260	0.5

The following aroclors can also be analyzed for upon request by the client, usually on a project basis. Since aroclors 1262 and 1268 contain peaks that elute much later than 1260, the GC run time needs to be extended to ensure the entire pattern has eluted.

Compound	LOQ (µg/L)
Aroclor 1262	0.5

Aroclor 1268	0.5
Aroclor 5422	0.5
Aroclor 5432	0.5
Aroclor 5460	0.5

See Appendix I for GC operating conditions and calibration information when analyzing for Aroclor 5422, 5432, and/or 5460.

Limits of Quantitiation (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

Basic Principles

A portion of an aqueous sample is extracted serially with methylene chloride. The volume of sample extracted can be adjusted depending on the physical appearance of the sample and the amount sent for analysis. The extract is dried, concentrated, and exchanged into hexane. The PCBs are then identified and quantitated using gas chromatography (GC) with an electron capture detector (ECD). The extract may require further cleanup (by florisil, sulfuric acid, or copper) to reduce matrix interferences that introduce large, unresolvable peaks in the chromatogram (due to interferents such as phthalates, oxygenated organics, unsaturated organics, or elemental sulfur).

Reference Modifications

Gas Chromatography conditions are different than those listed in Method 8082A however, all QC criteria are met.

Definitions

1. Analytical Batch – A group of field and Quality Control (QC) samples of the same matrix, extracted together under the same conditions and period of time, using the same lot(s) of chemicals.

2. Continuing Calibration Verification (CCV) – A mid-level standard used to verify that the analytical response is reliable, and has not changed significantly from the current Initial Calibration curve (ICAL). The verification of the ICAL that is required during the course of analyses at periodic intervals.

3. Initial Calibration Verification (ICV) – Second source calibration verification. A standard obtained or prepared from a source independent of the source of standards for the ICAL. Used to verify the integrity of the standards used for initial calibration.

4. Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD) – A sample of known composition analyzed with each batch of samples to demonstrate laboratory accuracy. The samples either naturally contain the analytes of interest or are clean samples fortified with known concentrations. Used to demonstrate laboratory accuracy. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – A sample created by fortifying a second aliquot of a water or soil sample with some or all of the analytes of interest. The concentration added is known and compared to the amount recovered to determine percent recovery. Matrix spike recoveries provide information about the accuracy of the method in light of the matrix analyzed. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

6. Method Blanks – A designated sample designed to monitor for sample contamination during the analysis process. A volume of deionized laboratory water is typically used to monitor water sample analysis, while solids blanks consist of a purified solid matrix or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

7. Surrogates – Organic compounds which are similar to the analytes of interest but are not naturally occurring in environmental samples. Surrogates are spiked into all standards and every field and QC sample prior to extraction and analysis to provide information regarding the effects of the sample matrix.

Interferences

A. Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

B. Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

C. An electron capture detector (ECD) is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.

D. Extracts may require further cleanup if interferents are present. Refer to 1-P-QM-PRO-9015477 (SOP-OE-004) for details on each cleanup procedure. Interfering materials can introduce large, unresolvable peaks into the chromatogram.

1. Use Florisil cleanup to reduce organics that can interfere (polar compounds).
2. Use GPC to remove sulfur and higher molecular weight organics.
3. Use copper or TBA cleanup to remove elemental sulfur.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Safety glasses must be worn around the GC where solvents and sample extracts are handled.

GC vials are disposed of in the designated lab container and subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the laboratory then taken to the lab-wide facility by personnel trained in hazardous waste disposal.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs. Refer to 1-P-QM-QMA-9015390 (LOM-SOP-ES-238) for more guidance on these options.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass containers with Teflon-lined caps, preserved with 0.008% sodium thiosulfate in case residual chlorine is present, and kept cool at 0° to 6°C, not frozen. Sample extraction must be performed within 7 days of collection, and sample analysis must be performed with 40 days of extraction.

Apparatus and Equipment

1. HP 7890 gas chromatograph equipped with an electron capture detector, or equivalent

2. Phenomonex MultiRes I – 30 m × 0.32 mm × 0.5 µm, or equivalent

3. Phenomonex MultiRes II – 30 m × 0.32 mm × 0.25 µm, or equivalent

4. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.

5. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents

1. Follow manufacturer's guidelines for storage conditions.

2. Hexane for autosampler rinse vials. Stored at room temperature.

3. UPC (Ultra Pure Carrier) helium for carrier gas.

4. UPC nitrogen for detector make-up gas.

5. UPC hydrogen carrier gas, either bottled or from a generator.

B. Standards

1. All standards are prepared using Class A volumetric pipettes, syringes, and flasks.

2. All weights are made on an analytical balance.

3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

4. All prepared standard solutions are stored at −10° to -15°C.

5. Aroclor 1016 stock – Restek 32006 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane.

6. Aroclor 1260 stock – Restek 32012 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane.

7. Aroclor 1221 stock – Restek 32007 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.

8. Aroclor 1232 stock – Restek 32008 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.

9. Aroclor 1242 stock – Restek 32009 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.

10. Aroclor 1248 stock – Restek 32010 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane. Two separate lots are kept on hand to provide a second source.

11. Aroclor 1254 stock – Restek 32011 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane. Two separate lots are kept on hand to provide a second source.

12. Surrogate stock (SS) – Supelco 861284 containing DCB/TCX at 200,000 ppb in acetone. Prepare an intermediate by diluting 0.25 to 25 mL of hexane.

13. 1016/1260 Matrix spike (MS) stock: Restek 32039. Each at 1,000,000 ppb in hexane. Also used as a second source stock. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

14. Aroclor 1262 stock – Restek #32409 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

15. Aroclor 1268 stock – Restek #32410 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

16. Instrument Blank (IBLK) – Surrogate Stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 200,000 ppb each in acetone.

17. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

d. For Bottle Code 43's

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
PCB Spike	1016/1260 MS Stock	1.25	250	Acetone or methanol	Water Spike
SS	SS Stock	1.5	1000	Acetone or methanol	SW-846 Water surrogate – identical to that prepared for Pest/PCB analyses

e. For Bottle Code 153's

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
Mini Sep. PCB Spike	PCB Spike	12.5	50	Acetone or methanol	Water Spike
SS	SS Stock	0.375	1000	Acetone or methanol	SW-846 Water surrogate – identical to that prepared for Pest/PCB analyses

18. An initial calibration verification standard must also be prepared at a concentration at the mid-point of the calibration for 1016, 1260, 1248, 1254 using a stock solution purchased from a different vendor or different lot than that used for the calibration standards.

19. Additional standards and preparations are listed in Appendix I for Aroclor 5442, 5432, and 5460 (PCT analysis).

Extraction

See organic extraction Analysis # 6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187

Gas Chromatographic Conditions

The conditions listed are usually the optimum operating conditions but can vary to improve the sensitivity, linearity, and overall chromatography or shorten run times on each GC system.

Detector	ECD
Detector Temperature	330°C
Oven Temperature	110°C

	40°C/min. to 250°C 20°C/min. to 280°C 30°C/min to 330°C Hold until DCB elutes ~ 2 min.
Column A	MR I
Column B	MR II
Carrier	Hydrogen at 12 psi, 5ml/min constant flow. (Can be substituted with Helium)
Makeup gas	N_2 at 80 mL/min. for Agilent GCs
Injection size	1 µL, direct injection
Injection Temperature	225°C

A Merlin microseal can be used in place of traditional septum.

Calibration

A. Prior to starting a new calibration, an analyst must change the septum on the GC and allow the system to stabilize (the septum change depends on the number of injections that have been made).

B. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.

C. Prepare a sequence using the following order of injections:

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR481
10. AR482
11. AR483
12. AR484
13. AR485
14. AR541
15. AR542
16. AR543
17. AR544
18. AR545
19. AR213
20. AR32x
21. AR423

22. AR623
23. AR683
24. MD16 (MDL)
25. IC16X (ICV)
26. IC48X (ICV)
27. IC54X (ICV)
28. Blank
29. LCS
30. 1234567
31. 1234567ms
32. 1234567msd
33.– 43. Continue running samples
44. AR163 (CCV)
45. IBLK

1. Note for our routine PCBs: The AR21 and AR42 are level 3's in the ICAL and are entered as AR213 and AR423 in the ICAL. AR32X is the only one that is an x and it runs at a level 3 concentration. Also the AR62 and AR68 are both level 3s and are listed as AR623 and AR683 in the sequences.

2. A single point of Aroclor 1016 is analyzed with the calibration to aid in better pattern recognition in the samples. Aroclor 1016 and 1242 have similar patterns with the exception of the smaller trailing peaks at the end of the Aroclor 1242 pattern. This is identified in the sequence as AR16xx.

3. A DDT/Endrin breakdown standard is run with each ICAL in order to provide the retention times of p,p -DDE, p,p-DDD, and p,p-DDT which may interfere with the pattern for aroclor 1254 and 1260.

4. See Appendix I for a sequence example when the analysis of PCTs (Aroclors 5442, 5432, and 5460) is requested.

D. Continue running groups of samples/injections. Each bracket must contain no more than 20 samples/injections and last no more than 12 hours for Method 8082A. Each bracket is followed by the continuing calibration check standard and an instrument blank (IBLK).

E. The conditioner injection is usually a standard or sample that has already been injected.

1. The conditioner is used to prime the system.

2. It is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

F. Hexane blanks can also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

G. Instrument blanks (IBLK) may also be run with the continuing calibration standards. This is optional but frequently requested for projects.

1. The instrument blank (IBLK) is injected after the conditioners but before the initial calibration.

2. It is used to determine that the instrument is free of background noise or contamination.

H. For projects where a known aroclor is present and at the request of clients other aroclors can be run for the continuing check standard. For instance, a set of continuing standards can be AR483, AR163, and IBLK.

I. Aroclor 1262 and 1268 can be analyzed when requested. A full five point curve can also be run, depending on the project requirements.

J. Initial Calibration (ICAL)

1. Calibrate for the aroclors using the five levels of 1016, 1260, 1248, and 1254. Use the single point for 1221, 1232, 1242, 1262, and 1268 when needed. As an option, 1248 and 1254 can use a single point calibration.

2 An external standard calibration based on the average calibration factor (AVG CF) for all analytes is used for quantitation where the %RSD is $\leq$20%.

3. The surrogate standards are calibrated using the AR16 levels using AVGCF unless the %RSD is >20%.

4. For the surrogates only: If the RSD is > 20%, use a calibration curve.

a. Use a linear fit.

b. The correlation coefficient must be >0.99 to be a valid fit.

c. Extrapolate or force to zero is not allowed. Set the zero to ignore. See 1-P-QM-PRO-9015498 (SOP-PP-031) for more details.

5. If the 0.99 curve coefficient cannot be met, or the 20% RSD for the aroclors:

a. Inspect the data points to see if one or more calibration level became concentrated due to solvent evaporation or degraded over time.

b. Reinject or remake the standard if a specific calibration level has concentrated due to solvent evaporation, or degraded over time..

c. Perform instrument maintenance as needed. See 1-P-QM-PRO-9015495 (SOP-PP-013) for troubleshooting linearity problems.

6. The calibration range for the aroclors is 50ug/L to 1000 µg/L. Level 1 is equivalent to the Limit of Quantitation.

7 Set up the aroclor calibration data in a custom program under Datalog.

The retention times of the peaks to use for identifying and quantifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations. See 1-P-QM-PRO-9015499 (SOP-PP-032) for details.

8. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections that contain them.

9. Set the scaling of chromatograms and peak integration parameters so that the size of the peaks for each compound of interest are approximately 2 to 3 mm in height at the concentration of the method detection limit (MDL).

a. Ensure all peaks in the MDL standard are integrated.

b. By running the 1016/1260 MDL standard, the majority of peaks of all aroclors are represented.

K. Initial Calibration Verification (ICV)

1. Verify the calibration curves using the ICV mixtures injected directly after the full ICAL.

2. The % difference of the concentrations for these must be within 20% difference of the nominal concentration.

a. If this criteria is not met, reinject the ICV.

b. If the criteria is not met again, then prepare a new standard.

L. Continuing Calibration Verification (CCV)

1. Calibration verification is performed after each set of twenty injections (samples, QC, blanks, etc.) or 12 hours, whichever comes first.

2. Use AR16 to evaluate the calibration of the aroclors (or other aroclors as requested for particular clients or projects).

3. Other aroclors can also be used along with AR16. This must be done if a site has prior history of containing a specific aroclor, or as requested for client projects or to meet other regulatory requirements.

4. The concentration quantitated for the continuing calibration check standards must be within 20% difference (%D) of the nominal concentration for each compound.

5. Samples must be bracketed with compliant standards.

a. Exception: If the standard following a sample is outside the ±20% but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected.

b. If continuing calibration checks continue to fail, corrective action must be taken, which can include performing injection port maintenance.

c. If confirmation of target analytes is needed, the second column should meet the 20% continuing calibration criteria as well as all initial calibration criteria.

6. The instrument blank (IBLK) is injected after each set of continuing calibration verifications when requested.

a. It must be evaluated as a water matrix against the water MDL/LOQs.

b. The IBLK must not have any target compounds above the reporting limits.

(1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be reinjected after another compliant IBLK.

(3) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up instrument.

7. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours or at no less than ±0.02 min, applied to the mid−point initial calibration standard.

b. If the RTs for a CCV fall outside the RT window, update the mid−point RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24−hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Make injections via an auto sampler.

2. Samples are analyzed according to the sequence in the calibration section above, along with the appropriate check standards.

3. Retention times of peaks in the samples are compared to the standard RT windows.

Peaks present on both columns that are also in the correct ratios to represent an aroclor are quantitated and the high value is reported unless chromatographic anomalies are observed. See 1-P-QM-PRO-9015494 (SOP-PP-011).

4. Use a minimum of three to six peaks for quantitation, with the exception of certain mixes where it can be more accurate to use less peaks to avoid excessive overlap of patterns.

5. If significant interference is present, schedule florisil and/or sulfuric acid cleanup. If elemental sulfur is present, copper treat the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract and adjust LOQs accordingly.

6. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

A. See 1-P-QM-PRO-9015501 (SOP-PP-040) for details on all calculations/equations used to evaluate the initial and continuing calibration.

B. Calculation of results is performed according to the following procedures:

1. The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each aroclor. Usually, the six major peaks that are unique to each aroclor are chosen for quantitation with the exception of 1221 where only three peaks are available.

2. Sample concentrations are calculated per peak using average calibration factor (AVG CF) from the initial calibration for 1016, 1260, 1248, and 1254 where a multi-point calibration is run, single point CF for 1221, 1232, 1242, (a multi-point calibration can also be run for these as requested or necessary to meet client or regulatory agency requirements). Do not use individual peaks that have values <MDL in quantitation.

3.

$$\frac{Sample\ Height}{AVG\ CF\ (CF)} \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

Where:

FV = Final volume – 10 mL

IV = Initial volume – 1000 mL

DF = Dilution factor, as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

4. The surrogate results are determined using either AVGRF or linear curve:

$$(Result\ 1 + Result\ 2 + \ldots + Result\ n) / n = Average\ Result$$

a. Using AVGCF from the initial calibration:

$$\frac{Sample\ Height}{AVG\ CF\ (CF)} \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

b. Using linear curve from the initial calibration:

$$[(Sample\ Height - Y - intercept)/Slope] \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

Where:

FV = Final volume – 10 mL

IV = Initial volume – 1000 mL

DF = Dilution factor, as needed

Statistical Information/Method Performance

Generate method detection limits (MDLs) and limits of quantitation (LOQs) according to (1-P-QM-QMA-9017309) LOM-SOP-ES-203. Perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire extraction and analysis procedure. Compare and pool results to determine the final reporting MDL. (NELAC allows for an annual verification of the MDL in lieu of an annual EPA MDL study).The department management maintains annual study data. The department manager requests that the QA Department update to the LIMS as needed. Update the department database via a download from the LIMS.

QC Acceptance limits are established as statistical limits. See 1-P-QM-PRO-9015496 (SOP-PP-025) for further information on monitoring and establishing limits.

Quality Assurance/Quality Control

A reagent water blank and a reagent water spike (LCS) are analyzed each day with every batch of samples (batch size is 20). An MS/MSD is performed per batch as long as there is ample volume of a sample in the batch. If an MS/MSD cannot be performed, an LCSD must be extracted.

Aroclor 1016 and 1260 are routinely spiked, however, other aroclors can be spiked as requested by clients.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

See 1-P-QM-PRO-9015493 (SOP-PP-002) for details on QC acceptance criteria and corrective action.

Appendix I

PCT Analysis (Aroclor 5442, 5432, and 5460)

A. Standards:

PCT-Stocks - Aroclor 5432, 5442, and 5460 purchased as individually ampulated solutions from Accustandard at 35,000ug/L.

Surrogate Stock - Ultra ISM-320 containing TCX/DCB at 200,000ppb in acetone. Prepare an intermediate by diluting 0.25mL to 25mL of hexane.

MS stock of Aroclor 5442 - Absolute Cat. #71791. 1000ug/L in Methanol.

Prepare working standards using the electronic standard database as a guide.

1. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

2. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

3. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

4. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
MS	5422 MS Stock	1.25	250	Acetone or methanol	PCT Water Spike
SS	SS Stock	1.5	1000	Acetone or methanol	PCB surrogate

B. GC Chromatographic Conditions

Detector: ECD
Detector temp: 300°C
Oven Temp: 110°C to 250° at 40°C/min, 280 to 330°C at 30°C/min, hold 9 min.
Carrier: Hydrogen at 5.0 mL/min
Makeup gas: N_2 at 80mL/min or equivalent
Injection size: 1-uL, direct injection
Injection temp: 225°C

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make any changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity, and/or improve separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

C. Sequence

1. Conditioner
2. IBLK
3. EVAL

4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR481
10. AR482
11. AR483
12. AR484
13. AR485
14. AR541
15. AR542
16. AR543
17. AR544
18. AR545
19. PCT1
20. PCT2
21. PCT3
22. PCT4
23. PCT5
24. A4421
25. A4422
26. A4423
27. A4424
28. A4425
29. AR213
30. AR32x
31. AR423
32. AR623
33. AR683
34. MD16
35. MDPCTX
36. IC16
37. IC48
38. IC54
39. Blank
40. LCS
41. 1234567
42. 1234567MS
43. 1234567MSD

44-58. Continue running samples

59. AR163
60. PCT3
61. A4423
62. IBLK

End of document

Version history

Version	Approval	Revision information
5	04.NOV.2015	

eurofins	Always check on-line for validity **Microwave Extraction Method 3546 for Pesticides in a Solid Matrix**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10926**		
Old Reference: **1-P-QM-WI -9015103**		
Version: **7**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **07-AUG-2017**	Document users: **5_EUUSLA_Organic Extraction_All, 6_EUUSLA_ Organic Extraction_Pest/PCB Soils, 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis DOD - 10496, 11141

Revision Log

Revision 7	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Basic Principles	Reflects current procedure	Added carbon cleanup.
Procedure 3	No longer part of this process	Removed reference to Mirex, MixE, and Kepone.
Procedure 14.c. (1)	Reflects current procedure	Added reference to carbon cleanup.

Revision: 6	**Effective Date:** Dec. 3, 2015	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Procedure	Reflect current procedure	Added pre-weighed sample process

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3546, Revision 0, February 2007.

2. MARS Operation Manual, Revision 2, February 2006.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #1363, 1420, 4225, 10738, 13237	Pesticides by Method 8081A in Solid Samples using GC-ECD
Analysis #2487	Food and Tissue Preparation
Analysis #10590	Analysis of Pesticides by 8081B in Solid Samples using GC-ECD
T-OE-PEST-WI11410	Pesticide Extract Cleanup Using Gel Permeation Chromatography
T-OE-PEST-WI10864	Glassware Cleaning for Organic Extractions
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-OE-PEST-WI10871	Pesticide Extract Concentration Using a Zymark TurboVap II Concentration Workstation
T-OE-PEST-WI10876	Organic Extraction Standards Storage and Handling

Scope

This procedure is used for the extraction of organochlorine pesticides from soils or solid wastes.

Basic Principles

A portion of sample is placed in an extraction vessel. Surrogate standards are added to each sample to monitor recovery. The vessel is then loaded into the instrument and extracted. The organic compounds present in the soil dissolve in the solvent, which is then removed. The sample is then concentrated and bottled.

Several cleanup procedures are used as needed to eliminate matrix interferences before the sample is analyzed. They include: florisil, copper, carbon cleanup, and gel-permeation cleanup (GPC).

Reference Modifications

The joint of the K-D is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

Definitions

1. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – A sample of known composition analyzed with each batch of samples to demonstrate laboratory accuracy. The samples either naturally contain the analytes of interest or are clean samples fortified with known concentrations. This is used to demonstrate laboratory accuracy. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

2. Matrix spike/matrix spike duplicate (MS/MSD) – A sample created by fortifying a second aliquot of a water or soil sample with some or all of the analytes of interest. The concentration added is known and compared to the amount recovered to determine percent recovery. Matrix spike recoveries provide information about the accuracy of the method in light of the matrix analyzed. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

3. Surrogates – Organic compounds which are similar to the analytes of interest but are not naturally occurring in environmental samples. Surrogates are spiked into all standards and every field and QC sample prior to extraction and analysis to provide information regarding the effects of the sample

Interferences

Method interferences are caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of sample to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.
The extracts are concentrated on a steam bath. Caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass jars with PTFE-lined lids and stored under refrigeration at 0º to 6º C, not frozen, prior to extraction.. Samples must be extracted within 14 days of collection. Extracts are stored in the freezer at -10° to -15°C.

Apparatus and Equipment

1. MARS Xpress – CEM Corp. or equivalent

2. Kuderna-Danish (K-D) assembly with appropriate ampule for concentrating the solvent used during concentration

3. Steam bath, VWR/LLI Model #1127 or equivalent

4. N-Evap with nitrogen supply

5. Beakers – stainless steel, assorted sizes

6. Pipettes – Class A, assorted sizes

7. Graduated cylinders – Class A, assorted sizes

8. Pipettes – disposable

9. Balance – capable of weighing to 0.01 g

10. Teflon®-wash bottles

11. Vials – assorted sizes

12. Teflon®-boiling chips

13. Forceps

14. Scoop

15. TurboVap II concentration workstation w/appropriate concentration tubes – Zymark or equivalent

16. Funnels – stainless steel or Teflon®

17. Extraction vessels

18. Frits – various

19. Sodium Sulfate Columns

20. GPC – J2 Scientific or equivalent

Reagents and Standards

1. Hexane – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt.

2. Acetone – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt.

3. Methylene Chloride (CH_2Cl_2) – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt.

4. Sodium Sulfate (Na_2SO_4) – Reagent grade or equivalent. Bake at 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

5. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical Analysis #1363, 1420, 4225, 10738, 13237 and #10590.

Preparation of Glassware

See T-OE-PEST-WI10864

Calibration

Not applicable to this procedure.

Procedure

1. Sample aliquot

a. If Sample Registration has pre-weighed the sample into a glass jar, add 5g of sodium sulfate, mix and proceed.

b. If the sample is not pre-weighed, weigh 30.0 - 30.5 g of sample into a labeled stainless steel beaker.

(1) Record the initial weight and any comments about the sample in the extraction log.

(2) Alternative sample weights may be used to meet certain reporting limits. However, if sample weight <30.5 is used the sample must be divided among multiple vessels and the extracts combined.

c. Process all tissues by Analysis #2487 prior to extraction.

d. The background, MS, and MSD are performed on three separate aliquots of a field sample.

e. Add 5 g of sodium sulfate to each sample and mix.

(1) If the sample has a high water content or is a clay-like soil, add an additional 10 g of sodium sulfate.

(2) Mix the sodium sulfate and sample until a free-flowing consistency is reached.

f. The Blank, LCS, and LCSD (if applicable) are prepared by filling a Teflon® extraction vessel with 35 g of sodium sulfate.

Record 30 g on the extraction log. (The sodium sulfate used for the QC samples is measured out as 35 grams to account for the 30 gram "sample" plus the 5 grams of sodium sulfate.)

2. Carefully place each sample into its clearly marked corresponding extraction vessel. A funnel is used to prevent spillage and loss of sample.

3. Use pipettes to add surrogate standards and spiking solutions.

a. Surrogates – 1.0 mL of SW-846 surrogate is added to all samples, blanks, and spikes.

b. Spiking solutions – Spiking solutions are added to the LCS, LCSD (if applicable), MS, and MSD samples. The type of spike is determined by an analysis number. Typically they are as follows:

(1) Spike 1.0 mL SW-846 spike.

Analysis #1420 has an additional LCS/LCSD spiked with 1.0 mL of Alachlor spike.

(2) These may change to accommodate specific client requirements as appropriate.

(3) See T-OE-PEST-WI10876 for storage and handling of spikes.

4. Add 30 mL of 50% acetone in methylene chloride to each vessel.

5. Cap each vessel according to manufacturer's guidelines.

6. Invert each vessel to ensure mixing of sample and solvent.

7. Place the vessels into the carousel. When all samples are loaded, place the carousel into the microwave.

8. Run Program "LL Soils". See Table 1 for instrument conditions. Verify that the run reached 100°C and document on the batchlog.

9. Uncap the cooled vessel.

10. Pour the extract and sample into a column filled with approximately 10cm of sodium sulfate on top of a Kuderna-Danish (K-D) assembly containing a Teflon®-boiling chip. Rinse the vessel with 10-20 mL of methylene chloride from a wash bottle.

11. Place a 3-Ball Snyder column on the K-D set-up, wet the column with methylene chloride, and concentrate over a steam bath at 85° to 99°C.

This steam bath temperature ensures concentration in a reasonable length of time.

12. **If the sample requires GPC, skip this Step.**

When the apparent volume in the ampule is 3 to 5 mL, use a graduated cylinder to add approximately 50 mL of hexane directly to the KD through the Snyder column.

Do not allow the ampule to go dry since loss of analytes will result.

13. When the apparent volume in the ampule again reaches 3 to 5 mL remove the sample from the bath and allow to cool for 10 minutes.

14. Remove the ampule and use a wash bottle to adjust the final volume.

a. Samples that do not require GPC: Adjust final volume to exactly 10 mL with hexane in a calibrated ampule. Mix thoroughly with a disposable pipette.

b. Samples that require GPC: Adjust the final volume to exactly 10 mL with methylene chloride. Mix thoroughly with a disposable pipette.

(1) Perform GPC cleanup following T-OE-PEST-WI11410

(2) When GPC cleanup is complete, concentrate the extract to final volume 5 mL using a TurboVap as described in T-OE-GEN-WI10871

c. Samples that require carbon cleanup: Adjust the final volume to exactly 10ml with hexane in a calibrated ampule. Mix thoroughly with a disposable pipette.

(1) perform carbon cleanup on the sample 2 mL to 2 mL as described in the Carbon Cleanup section of T-OE-PEST-WI10281

15. Florisil the sample 2 mL to 2 mL as described in the Pesticide Florisil Cleanup section of T-OE-PEST-WI10281

NOTE: DO NOT florisil samples that have Kepone as a compound of interest.

a. Bottle twice in an appropriately labeled crimp-top autosampler vial.

b. Place the remaining extract in an appropriately labeled screw-cap vial.

c. Store all extracts in the freezer at -10° to -15°C.

16. Perform copper cleanup upon request by the analytical department.

a. Removes sulfur interference from extracts.

b. See T-OE-PEST-WI10281 for this procedure.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
A batch is defined as the samples to be extracted in any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

For each batch of samples extracted, a blank, an LCS, MS, and MSD must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Table I

LL Soils: Instrument Conditions

Power: 1600W
Ramp Temperature: 100°C
Ramp Time: 30 minutes
Hold Time: 10 minutes
Cool Down Time: 20 minutes

End of document

Version history

Version	Approval	Revision information
6	03.DEC.2015	
7	04.AUG.2017	

An error has happened on file: /D4Doc/formularer/2016/09/Quality Document # 1-P-QM-WI -9015103_6_1049611141.doc
Errortext: The remote server returned an error: (404) Not Found.

eurofins	Always check on-line for validity **Analysis of Pesticides by 8081B in Solid Samples using GC-ECD**	Level: **Work Instruction**
Document number: **T-PEST-WI9232**		
Old Reference: **1-P-QM-WI-9015108**		
Version: **5.1**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **17-APR-2017**	Document users: **6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C**	Responsible: **5_EUUSLA_Pesticide Residue Analysis_Manager**

LIMS ID
Analysis DOD - 10590

Revision Log

Revision 5	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout document	Reflects current document numbers in D4	Replaced EtQ document numbers with D4 document numbers
Definitions	Basic terms defined in higher level documents	Removed section

Revision 5	**Effective Date:**	This version
Scope	Reflects current analysis	Added information for Pesticide in Tissue.
Scope	Clarification	Revised information in extraction and associated analysis scan table
Cross Reference	Reflects current cross reference Document and Document Titles	Updated document title for analysis #11129, 11131, 11134 Removed 1-P-QM-QMA-9015390 and 1-P-QM-QMA-9017309
Interference	Old reference not needed	Removed reference to SOP-OE-004
Personnel Training And Qualifications	Reference not needed	Removed reference to 1-P-QM-QMA-9015390
Sample Collection, Preservation, and Handling	Clarification	Added the extracts are stored at ≤-10ºC
Apparatus and Equipment	Reflects current columns	Added Phenomenex MR1 30 m × 0.32 mm × 0.5 µm and Phenomenex MR2 30 m × 0.32 mm × 0.25 µm
Reagents and Standards B.4.	Reflects current storage condition	Changed prepared storage conditions to ≤-10ºC
Reagents and Standards B.10.	Reflects current standard	Updated Chlordane standard from Ultra Scientific to Restek
Reagents and Standards B.13.	Reflects current standard	Updated EVAL standard from Restek to Ultra Scientific
Reagents and Standards B.15.	Relects current standard	Updated surrogate stock used for IBLK from Supelco to Restek
Reagents and Standards B.16.c.	Clarification	Added information for CCV, ICV, and MDL concentrations
Reagents and Standards B.16.d.	Clarification	Added detail to preparation of SS standard and MS standard
Calibration	Reflects current order of sequence	Changed the order of when the ICVs are injected
Calibration C.	Reflects current PCB standards	Changed the mixes that are injected for the PCB patterns
Calibration	Reflects current process	Removed statement on changing septum prior to ICAL
Calibration D. - L.	Reflects current process	Reordered steps throughout section.
Calibration J.3.c, e, J.5.d	Reference not needed	Removed references SOP-PP-031 and SOP-PP-013. Removed reference SOP-PP-032
Procedure 3	Reference not needed	Removed reference SOP-PP-011
Procedure 4	Reflects current process	Added information on evaluating data where compounds coellute on one of the columns.
Calculation	Reference not needed	Removed reference SOP-PP-040

Revision 5	**Effective Date:** This version	
Statistical Information/Method Performance	References not needed	Removed reference to 1-P-QM-QMA 9017309. Removed reference SOP-PP-025
Quality Assurance/Quality Control	Reflects current practice	Added kepone to list of compounds not routinely spiked
Quality Assurance/Quality Control	Reference not needed	Removed SOP-PP-002

Revision: 4	**Effective Date:** Oct 21, 2015	
Section	**Justification**	**Changes**
Scope	Reflect current compound list and LOQs	Updated compound list and LOQs
Sample Collection, Preservation, and Handling	Reflect current industry approach	Change to 0°to 6°C
Reagents and Standards	Clarification	Added statement on preparing spike and surrogate solutions
Calibration	Reflect current sequence	Updated sequence for TOX, Chlordane, and MIXE
Statistical Information/Method Performance	Clarification	Updated to same verbiage as the other Dept 24 analysis SOPs

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8081B, February 2007 (Update IV).

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #10496, 11141	Microwave Extraction Method 3546 for Pesticides in a Solid Matrix
Analysis #11129, 11131, 11134	Ultrasonic Extraction for Pesticides in a Solid Matrix by Method 3550
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-PEST-WI10011	QC Data Acceptability and Corrective Action
T-PEST-WI9954	Interpretation of Chromatographic Data
T-PEST-WI10007	Preventative and Corrective GC Maintenance
T-PEST-WI9980	Monitoring of QC Data Acceptance Limits
T-PEST-WI10016	Setting Up Single Component Initial Calibrations

Document	Document Title
T-PEST-WI10022	Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses

Scope

This method is used for identifying and quantitating the following Pesticides in solid samples by SW846 Method 8081B: Solid matrices other than soil can also be analyzed as long as the sample can be handled through the sonication or microwave technique for extracting. Typically solids are reduced to small pieces for extracting (e.g. concrete, wood, other plant material etc.). Tissue samples are ground and homogenized prior to extracting. Tissue samples may be whole fish, filets, or other miscellaneous species (usually aquatic, but not necessarily). Standards for the PCBs are run during this analysis since some of the PCB peaks may coelute or overlap with the pesticide peaks of interest. The information is used for proper identification and interpretation of the peaks observed for each sample. Quantitation of the same peak as a pesticide and PCB can be avoided.

Compound	LOQ (µg/kg)
alpha-BHC	0.83
beta-BHC	1.0
delta-BHC	0.90
heptachlor	0.83
aldrin	0.83
heptachlor epoxide	0.83
endosulfan I	0.83
endosulfan II	1.7
endosulfan sulfate	1.7
dieldrin	1.7
endrin	1.7
4,4'-DDE (*p,p*)	1.7
2,4'-DDE (*o,p*)	1.7
4,4'-DDD (*p,p*)	1.7
2,4'-DDD (*o,p*)	1.7
4,4'-DDT (*p,p*)	1.7
2,4'-DDT (*o,p*)	1.7
endrin aldehyde	1.7
endrin ketone	1.8
methoxychlor	6.7
chlordane	17
alpha-chlordane	0.83
gamma-chlordane	0.83
toxaphene	33.
kepone	7
mirex	1.7

Compound	LOQ (µg/kg)
telodrin	1.2
hexachlorobenzene(HCB)	0.83
Total Endosulfan (I + II)	0.83
Total DDTs	1.7

The following Pesticides in tissue samples are also identified and quantitated:

(**NOTE**: based on an initial sample volume of 15g to final volume 10mL)

Compound	LOQ (µg/kg)	LIMS Analysis Scan
alpha-BHC	1.7	13237
beta-BHC	3	13237
delta-BHC	5	13237
gamma-BHC (lindane)	5	13237
heptachlor	1.7	13237
aldrin	1.7	13237
heptachlor epoxide	1.7	13237
endosulfan I	1.7	13237
endosulfan II	3.4	13237
endosulfan sulfate	3.4	13237
dieldrin	3.4	13237
endrin	3.4	13237
4,4'-DDE (*p,p*)	3.4	13237
2,4'-DDE (*o,p*)	3.4	13237
4,4'-DDD (*p,p*)	3.4	13237
2,4'-DDD (*o,p*)	3.4	13237
4,4'-DDT (*p,p*)	3.4	13237
2,4'-DDT (*o,p*)	3.4	13237
endrin aldehyde	3.4	13237
endrin ketone	3.4	13237
methoxychlor	13	13237
Chlordane	34	13237
alpha-chlordane	1.7	13237
gamma-chlordane	1.7	13237
toxaphene	34	13237
mirex	3.4	13237
hexachlorobenzene(HCB)	2	13237

Limit of Quantitations (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

HCB, Telodrin, and Kepone, are additional compounds offered by special request.

The table below lists the extraction scan and the associated analysis scan.

Extraction	Analysis
10496 Microwave	10590
11134 Sonication	1363
10496 Microwave	13237

Basic Principles

The sample is extracted using sonication or microwave with 50% hexane/acetone. The extract is dried, concentrated, and exchanged to hexane. The pesticides are then identified and quantitated using gas chromatography with an electron capture detector (GC-ECD). Florisil, GPC, or copper cleanups may be employed to reduce matrix interferences which introduce large, unresolvable peaks into the chromatogram, specifically elemental sulfur.

Reference Modifications

Gas Chromatography conditions differ from those listed in 8081. However, all quality control (QC) criteria are met.

Interferences

A. Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

B. Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

C. An electron capture detector is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.

D. Extracts may require further cleanup if interferents are present. Refer to *T-OE-PEST-WI10281* for details on each cleanup procedure. Interfering materials can introduce large, unresolvable peaks into the chromatogram.

1. Use Florisil cleanup to reduce organics that can interfere (polar compounds).
2. Use GPC to remove sulfur and higher molecular weight organics.
3. Use copper cleanup to remove elemental sulfur.

E. For solid samples, a 1:1 mixture of methylene chloride/acetone has been shown to introduce many extraneous interfering peaks due to reactions that occur between the solvents during the heated concentration process. Therefore, hexane/acetone is used for extraction.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Lab coats and safety glasses must be worn around the GC where solvents and sample extracts are handled.

All GC vials and vials containing extracts are placed in a hazardous waste container for lab pack disposal. There is a satellite container in the laboratory that is then emptied into the main laboratory waste collection drums. All solvent waste is disposed of in solvent waste containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass containers with Teflon-lined caps and kept cool at 0° ± 6°C, not frozen. The extraction must be performed within 14 days of collection, and sample analysis must be performed with 40 days of extraction. Extracts are stored at ≤-10ºC.

Apparatus and Equipment

1. HP 6890 gas chromatograph equipped with dual electron capture detectors or equivalent

2. Columns:

   a. Phenomenex MR1 30 m × 0.32 mm × 0.5 µm

   b. Phenomenex MR2 30 m × 0.32 mm × 0.25 µm

Alternatively, Restek columns can also be used, and are in use for scan 1420 to achieve separation of alachlor

   c. RTX– CLPesticides – 30 m × 0.32 mm × 0.5 µm

   d. RTX – CLPesticides II – 30 m × 0.32 mm × 0.25 µm

3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.

4. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents

1. Hexane for autosampler rinse vials. Stored at room temperature for up to 1 year.
2. UPC (ultra pure carrier) helium for carrier gas
3. UPC nitrogen for detector make-up gas
4. UPC hydrogen for carrier, either bottled or from a generator

B. Standards

1. All standards are prepared using Class A volumetric pipettes, syringes, and flasks.

2. All weights are made on an analytical balance.

3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

4. All prepared standard solutions are stored at ≤ -10°C, except as noted.

5. See the electronic standards database for the compound list and concentrations contained in the various mixes.

6. PCB standards are identical to those outlined in analysis 0042 (1-P-QM-WI-9015126).

7. Mix A – Restek Catalog #32292. Equivalent to the CLP (SOW OLMO3.2) Mix A and B, contains all single component pesticides and surrogates in the TCL and PPL organochlorine lists.

8. Mix E – Restek Custom Mix #55992. Contains additional organochlorine pesticides such as kepone, the *o,p* isomers of DDT, DDD, DDE, mirex, telodrin and hexachlorobenzene.

9. Toxaphene stock – Restek Catalog #32005 at 1,000,000 ppb. Prepare an intermediate by placing 2 mL into a 25-mL volumetric and bring to volume with hexane.

10. Chlordane stock (CAS # 57-74-9) – Restek 32021(1,000,000 ppb in hexane).

11. Surrogate Stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetra-chloro-meta-xylene (TCX) at 200,000 ppb each in acetone.

12. Pest MS stock – Supelco Catalog #48796, #48196. All compounds in Mix A (#7 above)

13. EVAL stock – Ultra Scientific CLP-25D. Equivalent to CLP performance evaluation mix (PEM).

14. ICV stocks – These must always be from different lot numbers (or vendors) than the working calibration standards.

Accustandard Cat. #M-8081-SC and Accustandard Cat. #P-064S-10x for Methoxychlor. Prepare an intermediate by diluting 0.1 mL of stock into a 10 mL volumetric flask with hexane using the M-8081-SC stock.

15 Instrument Blank (IBLK) – surrogate stock (SS) – Restek 32000 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 200,000 ppb each in acetone.

16. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. MIXA1), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The standards are prepared in hexane. All standards are stored in glass bottles with Teflon lined caps in the freezer at ≤ -10ºC. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level. The CCV and ICV are at the level 3 concentration of the ICAL. The MDL standard is prepared at 2 to 5 times below the low level standard of the ICAL.

d. The following table is used for the preparation scheme of the matrix spike solution and the surrogate solution used during the extraction. The SS standard is prepared in a volumetric flask and stored in 8 separate 250ml clear glass bottles in the standard refrigerator at 0º to 6°C. The MS standard is prepared in a volumetric flask and stored in 9 separate 12ml amber vials in the standard refrigerator at 0º to 6°C.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	
SW-846 SS	SS Stock	3.0	2000	Aceto
SW-846 MS	MS Stock	2.0	100	Aceto

17. Standards for the PCBs are run during this analysis since some of the PCB peaks may coelute or overlap with the pesticide peaks of interest. The information is used for proper identification and interpretation of the peaks observed for each sample. Quantitiation of the same peak as a pesticide and PCB can be avoided.

Extraction

See Organic Extraction analysis 11131 (sonication) or analysis 10496 (microwave).

Gas Chromatographic Conditions

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity and/or improve the separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

Detector:	ECD
Detector	*Temp:* 330°C
Oven Temp:	140°C, no hold, 25°C/min to 250°C, then 22C/min to 300 C hold 3 min
Carrier:	Hydrogen at constant flow of 3.4 ml/min
	Helium may also be substituted
Makeup Gas:	N_2 at 55 mL/min
Injection Size:	1 µL, direct injection
Injector Temp:	225°C

Calibration

A. The pesticide analysis is run as a dual column approach. One injection is split onto two analytical columns. All initial and continuing calibration criteria listed below applies to both analytical columns.

B. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.

C. Prepare a sequence using the following order of injections:

1. Conditioner
2. IBLK (instrument blank)
3. EVALX
4. MIXA1
5. MIXA2
6. MIXA3
7. MIXA4
8. MIX A5
9. ICMAX (ICV STD)
10. MIXE1*
11. MIXE2*
12. MIXE3*
13. MIXE4*
14. MIXE5*
15. ICMEX* (ICV if needed)
16. TOXA1+ **(if one level is run use TOXA1; if 5 levels are run use TOXA1-TOXA5)**
17. ICTX* (ICV if needed)
18. CHLD3+ **(if one level is run use CHLD1; if 5 levels are run use CHLD1- CHLD5)**
19. ICCH* (ICV if needed)

20. AR163
21. 2154X
22. AR483
23. MDLA (MDL std for Mix A)
24. MDLE (MDL std for Mix E)
25. MDTX (MDL std for toxaphene, only if 5 level TOX curve is run)
26. MDCH (MDL std for chlordane, only if 5 level CHLD curve is run)
27. Blank
28. LCS
29. 1234567
30. 1234567ms
31. 1234567msd
32.– 42. Continue running samples for a 12-hour period from last standard
43. EVALX
44. MIXA3 (CCV)
45. MIXE3* (CCV)

D. The conditioner injection is usually a standard or sample that has already been injected.

1. The conditioner is used to prime the system

2. It is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

E. Hexane blanks can also be injected to allow the GC to go through some temperature programs and/or check the cleanliness of the system.

F. Inject initial instrument blank (IBLK) after the conditioners but before the initial calibration. It is used to determine that the instrument is free of background noise or contamination.

G. Run a breakdown evaluation standard (EVAL) at the start of an ICAL to ensure the breakdown of DDT and endrin meets the method acceptance criteria.

1. Sample analysis cannot be performed if the breakdown exceeds 15% for either DDT or endrin.

2. Breakdown check is run ongoing throughout the run after every 12 hours.

3. The sum of the peak heights of DDE, and DDD divided by the total peak heights of DDT/DDD/DDE cannot exceed 15%.

4. The sum of the peak heights endrin aldehyde and endrin ketone divided by the total peak heights of endrin/endrin aldehyde/endrin ketone cannot exceed 15%.

H. *MIX E is only needed when analyzing for additional pesticide including 2,4-DDE, 2,4-DDD, 2,4-DDT, kepone, mirex, HCB and telodrin.

I. The order of injections for the multi-components is not critical as long as they are all run before sample analysis.

1. Aroclors are only used to identify possible PCB patterns which may interfere with the pesticide detections.

2. A single level of toxaphene and chlordane can be run for use in identifying the presence of these target compounds; however,

a. If either of these compounds are detected in a sample, the sample must be rerun with a full five-point curve.

b. The full curve can be run at the outset of a new calibration if the samples to be run are known to contain toxaphene or chlordane.

J. Initial Calibration (ICAL)

1. Calibrate using the 5 levels of the single component pesticides contained in MIX A and E, and using the single point for chlordane, toxaphene (or a full curve when sample(s) contain toxaphene or chlordane).

2. An external standard calibration is used with average calibration factor (AVGCF) for all analytes where the %RSD is ≤20%.

3. If the RSD is > 20%, use a calibration curve.

a. Attempt a linear fit first. Use this fit if the correlation coefficient is ≥0.99.

b. If the correlation coefficient is less than 0.99, a quadratic fit will be tried.

(1) A six-point calibration must be run to use quadratic.

(2) Prepare a sixth point somewhere within the established calibration range listed in the standards preparation section.

(3) Typically the pesticides in this method will not require a quadratic fit.

(4) Quadratic fit can not be used to extend the calibration range or bypass instrument maintenance

NOTE: For samples from ***South Carolina***, a quadratic fit may not be used.

c. For either curve type, extrapolate or force to zero is not allowed. Set the zero to ignore. See *T-PEST-WI10016* for more details.

d. If toxaphene or chlordane is detected in a sample, the sample will be rerun along with a full five−point calibration for that analyte as well as check standards.

e. If the 0.99 curve coefficient cannot be met:

(1) Inspect the data points to see if one or more calibration levels appear to be off.

(2) Reinject or remake the standard if a specific calibration level has concentrated due to solvent evaporation, or degraded over time.

(3) Perform instrument maintenance as needed. See *T-PEST-WI10007* for troubleshooting linearity problems.

f. Curve types and criteria can be altered to meet client or project specific requirements as well as any regulatory agency requirements that may differ from those listed here.

4. Set up the aroclor data in the custom “datalog” program.

a. The retention times of the peaks used for identifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations.

b. This calibration is only used to help identify potential aroclor patterns and peaks that may overlap or interfere with target pesticides.

5. Set up the toxaphene and chlordane calibration data in the custom “datalog” program.

a. The retention times of the peaks used for identifying and quantifying these multi-component pesticides are entered into the calibration file along with the corresponding peak heights and concentrations.

b. The calibration is external standard using the AVGRF.

c. Toxaphene and chlordane must meet the 20% RSD criteria.

d. See **T**-PEST-WI10022 for details on this program.

6. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections that contain them.

7. Set the scaling of chromatograms and peak integration parameters so that the peaks for each compound of interest are detected and integrated at the concentration of the MDL. This ensures that the LOQ and MDL can be met.

K. Initial Calibration Verification (ICV)

1. Verify the calibration curve using the ICV mixtures injected directly after the corresponding curve.

2. The % difference of the concentrations for these must be within 20% difference of the nominal concentration.

L. Continuing Calibration Verification (CCV)

1. Analyze a set of check standards after each set of injections in a 12-hour period, or 20 samples, whichever comes first (samples, QC, blanks, etc.).

a. Use a mid level single component standard to evaluate the response of single component analytes.

b. Run the breakdown evaluation mix (EVAL). Analysis cannot proceed if either compound exceeds 15%.

2. The concentration quantitated for the continuing calibration check standards must be within 20% difference (%D) of the nominal concentration.

3. Samples must be bracketed by compliant standards.

4. When confirmation of target analytes is needed, the initial calibration criteria must be met and the second column should meet the 20% continuing calibration criteria.

5. When a continuing calibration verification standard (CCV) fails to meet the QC criteria, all samples that were injected after the last CCV that passed must be re-injected.

Exception: If the CCV fails high and those targets are not detected in the associated samples, the samples can be reported.

6. The instrument blank (IBLK) may be injected after each set of continuing calibration verifications.

a. This is optional but frequently requested for projects.

b. It must be evaluated as a water matrix against the water MDL/LOQs.

c. The IBLK must not have any target compounds above the reporting limits.

(1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be reinjected

(3) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up the instrument.

7. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours, or at no less than .02 minutes, applied to the initial calibration standard, usually Level 3.

b. If the RTs for a continuing calibration standard fall outside the RT window, update the midpoint RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24−hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Make injections via an auto sampler.

2. Samples are analyzed according to the sequence in the calibration section above.

3. Retention times of peaks in the samples are compared to the standard RT windows. Peaks present on both columns (and that are also in the correct ratios to represent an aroclor) are quantitated and the high value is reported unless there are chromatographic anomalies. See *T-PEST-WI9954*.

4. There are several compounds that coelute on one column and not the other; whether a compound coelutes or not is instrument and column dependent. Compounds known to coelute are contained in different standards. One standard is named MIXA and the other standard is MIXE. Refer back to the ICAL to note which compounds coelute and on which column they coelute before evaluating the sample data. If there is a detection for one of the coeluting compounds on the column where this compound does not coelute and the detection is greater than the MDL, look at the other column and note if the coeluting compound is present. If the coeluting column is present and within the +/-0.02 retention time units of the detected coleluting compound from the other column, calculate the concentration using the

appropriate response factor. Calculate the RPD between the two columns and report as per routine protocol.

5. Continue running groups of samples/injections followed by check standards every 12 hours or every 20 injections, whichever comes first.

6. If significant interference is present, schedule florisil cleanup. If elemental sulfur is present, copper cleanup the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract, analyze, and adjust the LOQs accordingly.

7. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

A. See *T-PEST-WI9847* for details on all calculations/equations used to evaluate the initial and continuing calibration.

B. Calculation of results is performed according to the following procedures:

1. Single-component compounds

   a. Using AVGCF from initial calibration

$$\frac{Sample\ Height}{AVG\ CF\ (CF)} \times \frac{FV}{IW} \times DF = \mu g/kg\ as\ received$$

   b. Using linear curve from initial calibration:

$$[(Sample\,Height - Y - intercept)/Slope] \times \frac{FV}{IW} \times DF = \mu g/kg\,as\,received$$

Where:

FV (final volume) = volume in mLs
IW (initial weight) = weight in grams

DF (dilution factor) = as needed

2. Multi-component compounds

The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each quantitation peak used for toxaphene and chlordane.

Usually the six major peaks are chosen for quantitation. Include alpha chlordane, gamma chlordane, and heptachlor as three of the six major peaks for the calibration of chlordane. Sample concentrations are calculated per peak using Average Calibration Factor (AVG CF).

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{FV}{IW} \times DF = \mu g/kg \text{ as received}$$

Where:

FV (final volume) = volume in mLs

IW (initial weight) = weight in grams

AF (additional factor) = based on cleanups
(i.e., 2 mL extract florisiled to 25 mL = 12.5)

DF (dilution factor) = as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(\text{Result 1} + \text{Result 2} + \ldots + \text{Result n}) \; / \; n = \text{Average Result}$$

NOTE: If toxaphene or chlordane is detected in a sample, the sample will be rerun along with a full five-point calibration for that analyte and a check standard.

3. A breakdown mix (EVALX) containing p,p′-DDT and Endrin is run to check for breakdown. The breakdown must not exceed 15% for either compound. Breakdown is calculated as:

$$\frac{\%\ \text{Breakdown}}{\text{for p,p'-DDT}} = \frac{\text{pk ht (area) of p,p'-DDE + p,p'-DDD}}{\text{pk ht (area) of p,p'-DDE + p,p'-DDD + p,p'-DDT}} \times 100$$

$$\frac{\%\ \text{Breakdown}}{\text{for Endrin}} = \frac{\text{pk ht (area) of Endrin Aldehyde + Endrin Ketone}}{\text{pk ht (area) of Endrin Aldehyde + Endrin Ketone + Endrin}} \times 100$$

If breakdown fails, injector maintenance must be performed. Analysis cannot proceed until breakdown check passes.

Statistical Information/Method Performance

Initially, perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire extraction and analysis procedure. Compare and pool results to determine the final reporting MDL. An MDL study or verification of the MDL is required each year. NELAC allows for an annual verification of the MDL in lieu of a full MDL study. The department management maintains annual study data. The department manager requests that the QA Department update to the LIMS as needed. Update the department database via a download from the LIMS.

QC Acceptance limits are established as statistical limits. See *T-PEST-WI9980* for further information on monitoring and establishing limits.

Quality Assurance/Quality Control

Each extraction batch (up to 20 samples) must contain a method blank, a laboratory control spike sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD) or an LCS/LCSD.

The TCL single-component pesticides of interest for each analysis are routinely spiked. Mirex, *o,p*−DDE/DDD/DDT, telodrin, kepone, and HCB are not spiked since this would result in co−elution with the other spiked compounds. These can be spiked at a client's request for special projects, within our scope of accreditation.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

QC limits for surrogates, LCS/LCSD, and MS/MSD are established through statistical analysis of historical data. The limits are evaluated every 6 months and updated as needed. The limits are maintained in the LIMS for the relevant analysis numbers.

See *T-PEST-WI10011* for details on QC acceptance criteria and corrective action.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

T-PEST-WI10007 Preventative and Corrective GC Maintenance
T-PEST-WI10011 QC Data Acceptability and Corrective Action
T-PEST-WI10016 Setting Up Single Component Initial Calibrations
T-PEST-WI10022 Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PEST-WI9954 Interpretation of Chromatographic Data
T-PEST-WI9980 Monitoring QC Data Acceptance Limits

End of document

Version history

Version	Approval	Revision information
4	21.OCT.2015	
5	17.FEB.2017	
5.1	17.APR.2017	

eurofins	Always check on-line for validity **Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10920**		
Old Reference: **1-P-QM-WI -9015079**		
Version: **19**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **07-OCT-2016**	Document users: **5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Pest/PCB Waters, 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis DOD - 6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187

Revision Log

Revision: 19		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects current analysis numbers	Added Analysis #13634, 14134, 14166, 14169, 14184, 14186, 14188
Apparatus and Equipment	Reflects current practice	Added Automated Water Extraction Bench, Rapid-Vap Evaporator, and Rapid-Vap Evaporator Tube

Revision: 19		**Effective Date:** This version
Procedure 6.b. & 6.c.	Reflects current practice	Added surrogate and spike details for new analysis numbers
Procedure 11-19	Reflects current practice	Separate 1L extraction from 250 mL extraction and added new procedure for 250 mL extraction

Revision: 18		**Effective Date:** Apr 13, 2016
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Apparatus and Equipment	Reflects current process	Added Turbo Vap tubes and Turbo Vap
Procedure 13	Reflects current process	Added 13.b for analyses that require Turbo Vap concentration
Procedure 18	Reflects current process	Added 18.b for analyses that require Turbo Vap concentration
Procedure 19	Reflects current process	Added 19.b, 19.c, and19.d to differentiate between which steps require Turbo Vap concentration and which steps require steam bath concentration.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3510C, Rev. 3, December 1996.
2. USEPA, 40 CFR Part 136, Appendix A, Method 608.
3. USEPA, 40 CFR Part 136, Appendix A, Method 622.
4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0177, 0950, 0180, 1954	Pesticides in Water by Method 8081A using GC-ECD
Analysis #2257, 2253	Captan and Captafol by Method 8081A in Waters and Solids using GC-ECD
Analysis #5366, 10410, 10593, 12144, 13182, 13186	Organophosphorous Pesticides by Methods 8141A/8141B/622 in Aqueous Samples using GC-NPD
Analysis #6030, 10227	Polychlorinated Biphenyls (PCBs) by Method 608 or 8082 in Waters
Analysis #7572	Pesticides in Aqueous Samples by Method 608
Analysis #10589, 10647	Pesticides in Water by Method 8081B using GC-ECD
Analysis #10591, 13092	Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples using GC-ECD
	Low Level PCBs in Water by Method 8082/8082A using GC-ECD

Analysis #12013, 12686	
1-P-QM-PRO-9015407	Pesticide Extract Cleanup Using Gel Permeation Chromatography
1-P-QM-PRO-9015475	Glassware Cleaning for Organic Extractions
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Purpose
The purpose of this SOP is to provide clear instructions for performing the separatory funnel extraction procedure on samples that are to be analyzed for pesticides and PCBs.

Scope
This procedure is for the extraction of organochlorine and organophosphorous pesticides and PCBs from wastewaters.

Basic Principles
An aliquot of the sample is placed into a separatory funnel. The volume of sample extracted is adjusted (if appropriate) depending on the physical appearance of the sample and the volume sent for analysis. A surrogate standard is added to the sample to monitor recovery. The sample is then extracted with methylene chloride. The extract is dried, concentrated, and exchanged to hexane. Several cleanup procedures are available to eliminate matrix interferences before the sample is analyzed. They include sulfuric acid treatment, copper treatment, florisil, and Gel-Permeation Cleanup (GPC).

Reference Modifications
1. Surrogate and matrix spiking solutions are not added before the transfer to the extractor. For several reasons:

a. Samples must be poured from the amber bottles to determine the matrix and volume of sample to use for each extraction.

b. Many sample bottles have no headspace and there is no room to add surrogate to the sample in the bottle.

c. Due to the volume of samples extracted, a separate graduated cylinder for each sample is unrealistic.

d. To maintain consistency with all extractions, no samples are spiked in the bottle or graduated cylinders.

2. In Procedure Step 19 the joint of the KD is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

Interferences

Impurities in solvents, reagents, glassware, or other hardware used in sample processing interfere with the method. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the employees training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the extraction and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, or one blind sample.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass bottles with PTFE-lined lids, preserved with sodium thiosulfate, and stored refrigerated at 0 - 6°C, not frozen. Sample extraction must be started within 7 days of collection. Extracts are stored frozen at ≤-10°C.

Apparatus and Equipment

1. Separatory funnel for extracting organic components from an aqueous matrix

2. Kuderna-Danish (K-D) assembly with appropriate ampule for extracting the solvent used during the extraction

3. Water bath – VWR/LLI Model #1127 or equivalent

4. Sodium sulfate column with extra course frit

5. Nitrogen evaporation (N-Evap) with nitrogen supply – Organomation Associates or equivalent

6. pH paper – Wide range

7. Automatic shaker – Glass Col or equivalent, capable of holding 2-L separatory funnels

8. Pipettes – Class A, assorted sizes

9. Graduated cylinders – Class A, assorted sizes

10. Pipettes – Disposable

11. Solvent dispenser – Brinkmann, adjustable or equivalent

12. Balance – Capable of weighing to 0.01 g

13. Centrifuge – Beckman GS-6 or equivalent

14. Micro-Snyder columns

15. Wash bottles – Teflon

16. Vials – Assorted sizes

17. Teflon boiling chips

18. Syringes - Assorted sizes

19. Micro-pipetter

20. Turbo Vap – Zymark Turbo Vap II concentration station or equivalent

21. Turbo Vap tubes- 250ml Zymark tubes or equivalent

22. Automated Water Extraction Bench – capable of holding separatory funnels for 250 mL extractions.

23. Rapid-Vap Evaporators – for concentration of 250 mL extractions

24. Rapid-Vap Evaporator Tubes – Class A

Reagents and Standards

1. Methylene chloride (CH_2Cl_2) – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

2. Acetone – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

3. Hexane – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

4. 10N Sodium hydroxide (NaOH) – Lab Chem or equivalent. Store at room temperature. Follow manufacturer's expiration date.

5. Sulfuric acid (H_2SO_4) – ACS grade or equivalent. Store at room temperature. Follow manufacturer's expiration date.

6. Sodium Sulfate (Na_2SO_4) – Reagent grade or equivalent. Bake at approximately 400°C for a minimum of 4 hours in a shallow pan to remove organic contaminants. Store in a glass jar at room temperature for up to 1 year after baking.

7. Reagent water – water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the reagent water supplied at the taps in the laboratory meets this criterion. If the reagent water does not meet the requirements, see your supervisor for further instructions.

8. Extraction fluid – Prepared and delivered by the leachate department. Store refrigerated at 0-6^{o}C, not frozen, in a glass container with a PTFE-lined lid.

9. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical
Analysis #0177, 0950, 0180, 1954,14134; Analysis #2257, 2253; Analysis #5366 10410, 10593, 12144, 13182, 13186; Analysis #6030, 10227, 14169, 14188; Analysis #7572, 13634; Analysis #10589, 10647, 14166; analysis 10591, 13092, 14184, 14186 ; and Analysis #12013, 12686.

Calibration

Not applicable to this procedure.

Preparation of Glassware

See SOP 1-P-QM-PRO-9015475.

Procedure

1. Determine the volume of sample to be used for each extraction. Typically, this is 1L for routine extractions or 250mL for 250mL extractions.

1 Liter Analyses: 0177, 0180, 1954, 2257, 2253, 5366, 10410, 10593, 12144, 13182, 13186, 6030, 10227, 7572, 10589, 10647, 10591, 13092, 12013, 12686

250 mL Analyses: 13634, 14134, 14166, 14169, 14184, 14186, 14188

a. If uncertain of the volume to extract for any sample, ask your supervisor.

b. Use one full bottle for all analysis scans (with the exception of scan #0950) unless the matrix is poor (thick, lots of sediment, extremely foul odor).

(1) If using reduced volume due to matrix, take as much as possible while trying to minimize matrix problems, document why the reduced volume was used.

(2) Reduced volume aliquots due to matrix are typically are 500, 200, or 100 mL.

c. For analysis 0950:

(1) If the sample bottle contains at least 500 mL of sample, measure 200 mL.

(2) If the sample bottle contains <500 mL of sample, use 1/2 of the available volume or 10 mL, whichever is greater.

(3) If a matrix spike (MS) or MS/matrix spike duplicate (MSD) is required for the sample, use 200 mL each.

d. The background, MS, and MSD are performed on three separate aliquots of a field sample.

2. Prepare the blank, laboratory control sample (LCS), laboratory control sample duplicate (LCSD) (if applicable) with 1 L of reagent water (or 250mL for 250 mL extractions) measured into the separatory funnel.

Exception: For Analysis #0950 - The blank, LCS, and LCSD (if applicable) are prepared using 200 mL of extraction fluid measured into the separatory funnel.

3. For samples using 1 entire bottle:

a. Etch the outside of the bottle with a scriber at the meniscus.

b. Shake the bottle vigorously and then pour the contents into a separatory funnel.

4. For all samples requiring a specified volume:

a. Shake each bottle vigorously.

b. Use a clean graduated cylinder to measure the desired volume.

c. Use a wash bottle to rinse the graduated cylinder with methylene chloride and add the rinsate to the separatory funnel.

d. If <1000 mL (or <250mL for 250mL extractions) of sample is used, use a graduated cylinder to add enough reagent water to bring the volume in the extractor to 1−L (or 250mL for 250mL extractions).

5. Record any comments about the sample and the initial volume, if known at this time, on the extraction sheet.

6. Use pipettes to add surrogate standards and spiking solutions to the aqueous sample in the separatory funnel.

a. Be certain the standard drips directly into the aqueous sample without touching the glass side of the separatory funnel to avoid poor recoveries.

b. Surrogates – Surrogates are added to all samples, blanks, and spikes.

The type of surrogate is determined by the analysis scan number. Typically they are as follows:

(1) For analyses 0177, 0180, 0950, 1954, 2257, 6030, 7572, 10589, 10591, 13092, 10647, 13092 – 1.0 mL SW−846 Surrogate.

(2) For analysis 5366, 10593, 10410, 12144, 13182, 13186 – 1.0 mL NP Surrogate

(3) For Analysis #12013, 12686 – 0.1 mL of SW-846 Surrogate

(4) For Analysis #10227 – If entered for prep 11117 use 1.0 mL of SW846 Surrogate. If entered for prep 13086 use 1.0 mL of 2mL SW846 Surrogate.

(5) For Analyses #13634, 14134, 14166, 14169, 14184, 14186, 14188 – Use 1.0 mL of Mini SW846 Surrogate

c. Spiking Solutions – Spiking solutions are added to the LCS, LCSD (if applicable), MS, and MSD.

(1) The type of spike is determined by an analysis number. Typically they are as follows:

(a) For analysis 6030, 10591, 13092 – 1.0 mL PCB Spike

(b) For analysis 10227 – If entered with prep 11117 use 1.0 mL of PCB spike. If entered with prep 13086 use 1.0 mL of 2mL PCB spike.

(c) For analysis 7572, 10589, 0177 – 1.0 mL SW-846 Spike

(d) For analysis 0950 & 10647 – 1.0 mL TCLP Pesticides Spike regardless of initial volume

(e) For analysis 5366 – 1.0 mL Triazine Herb Spike

(f) For analyses 10593 and 10410 – 1.0 mL OPPA Spike

(g) For analysis 2257 – 1.0 mL Captan/Captafol Spike

(h) For analysis 0180 – 1.0 mL of Chlordane spike

(i) For analysis 1954 – 1.0 mL of SW846 spike and an additional LCS/LCSD with 1.0 mL of Alaclor

(j) For analysis 12013 – 0.1 mL of PCB Spike

(k) For Analysis 13093 – 1.0 mL of PCB Spike and prepare a separate LCS/LCSD using 1.0 mL of 5442 Arochlor Spike

(l) For Analysis 13182 and 13186 – 1.0mL of Appendix IX Water Spike (or 0.25 mL of Appendix IX water Spike for 250mL extractions).

(m) For analysis 13634, 14134, 14166 – 0.25 mL of SW846 Spike

(n) For analysis 14169, 14184, 14188 – 1.0 mL of Mini PCB Spike

(o) For analysis 14186 – 1.0 mL of Mini PCB Spike and prepare a separate LCS/LCSD using 1.0 mL of Mini 5442 Arochlor Spike

(2) Spike details can be found in the corresponding analytical SOP(s)

(3) This is changed to accommodate specific-client requirements, if appropriate.

(4) If a sample requires any special compounds in addition to the standard list, an appropriate spike containing those compounds is added at this time.

(5) See SOP 1-P-QM-PRO-9015490 for storage and handling of spikes.

7. Measure and record the pH of the sample using wide-range pH paper.

a. If necessary, adjust the pH to between 5 and 9 using 10N NaOH (to bring up the pH) or concentrated H_2SO_4 (to lower the pH).

b. To adjust the pH, add a few drops of the appropriate solution with a disposable pipette, shake the separatory funnel and re-check the pH.

c. If the pH now falls between 5 and 9, record the adjusted pH on the extraction sheet.

d. If the pH is still out of range, continue adding small aliquots of base or acid, shaking and checking the pH until it is within the specified range.

e. Record the adjusted pH.

NOTE: Any samples from North Carolina require that the volume of acid or base added to the sample to adjust the pH to be recorded on the extraction sheet.

8. If the original sample bottle is empty:

a. Use a solvent pump to measure 60 mL (15 mL for 250 mL extractions) of methylene chloride. Add the methylene chloride to the sample bottle. Then cap the bottle and invert several times. Add the solvent to the separatory funnel.

b. Fill the bottle to the marked level with water and transfer the water to a graduated cylinder to determine the initial volume.

c. Alternatively, for all analyses **except Analysis #7572, and analysis 6030 (Method 608)** weigh the empty bottle and tare the balance.

(1) Fill the bottle to the marked level with water and place the bottle onto the tared balance.

(2) This weight rounded to a whole number is the initial sample volume.

d. Record the initial volume on the extraction sheet.

9. If the sample container is not empty, measure 60 mL (15 mL for 250mL extractions) of methylene chloride and add the solvent directly to the separatory funnel.

10. Cap the funnel, invert it, and vent immediately.

11. For 1 Liter Analyses: Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

For 250 mL Analyses: Separatory funnels remain on the Automated Water Extraction Bench. Press the green “start” button to lower the hood and activate the Automated Water Extraction Bench. The bench will tumble for 2 minutes. The sash will rise after the samples have tumbled.

NOTE: Shaker speeds vary greatly between instruments so the proper setting is marked on each.

12. For 1 Liter Analyses: Place the separatory funnel on the rack and allow it to sit undisturbed for approximately 10 minutes. For 250 mL Analyses: the separatory funnel will sit undisturbed on the Automated Water Extraction Bench.

a. The time required for extracts to set undisturbed is based upon visual confirmation that the layers are adequately separated. Additional time may be necessary for samples with unusually high density (i.e. high salt content).

b. If an emulsion forms and is greater than 1/3 of the volume of the solvent layer, mechanical techniques such as stirring and centrifugation must be employed to complete the separation.

13 Removing the Solvent Layer.

a. For 1 Liter analyses (except 5366, 10593, 10410, 12144, 13182, and 13186) : Remove the solvent layer by opening the stopcock and collect the solvent in a metal beaker then pour through approximately 10cm of sodium sulfate into a K-D apparatus containing a Teflon boiling chip.

b. For analyses 5366, 10593, 10410, 12144, 13182, and 13186, remove the solvent layer by opening the stopcock and collecting the solvent in a metal beaker. The solvent layer is then poured into a sodium sulfate column and collected in a Turbo Vap tube.

c. For 250 mL analyses: drain the solvent layer directly into the sodium sulfate column containing approximately 5cm of sodium sulfate. This drains into a Rapid-Vap Evaporator Tube.

NOTE: Do not use more than the recommended quantity of sodium sulfate and take care not to transfer water into the column. Excessive sodium sulfate and/or water in the column results in low aldrin recovery.

14. Use a solvent pump to add 60 mL (15mL for 250mL extractions) of methylene chloride to the separatory funnel and repeat Procedure Steps 10 through 13, venting only as necessary.

15. Again, use a solvent pump to add 60 mL (15 mL for 250mL extractions) of methylene chloride to the separatory funnel and repeat Procedure Steps 10 through 13, venting only as necessary.

16. For 1L extractions only, rinse the metal beaker that was used for solvent collection with approximately 20 mL of methylene chloride. Pour into salt column.

17. Rinse salt column with approximately 20 mL (5mL for 250mL extractions) of methylene chloride. Use a hand held bulb to squeeze through any excess methylene chloride.

18. Concentrating the Extract.

a. For 1 Liter Analyses: Attach a 3−ball Snyder column to the K−D, wet with solvent, and concentrate the extract to approximately 3 to 5 mL on a steam bath at 85° to 99°C. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. This steam bath temperature ensures concentration in a reasonable length of time.

b. For 250 mL Analyses: Place the Evaporator Tube containing the solvent extract into the pre-heated (approximately 80°C) Rapid-Vap Evaporator. Set the timer for 15 minutes and speed at 75% (the speed is the rotation within the unit while the sample is evaporating). Confirm that the nitrogen is set at 15 psi (this is adjusted on the regulator attached to the nitrogen line). Allow samples to concentrate to between 1-2mL.

Note: The temperature, timer, and speed settings can be adjusted as necessary to accommodate difficult sample matrices, etc.

c. For analyses 5366, 10593, 10410, 12144, 13182, and 13186, place the Turbo Vap tubes in a Turbo Vap with the temperature set between 45°and 50°C.

To avoid loss of analytes do not allow the ampule or Turbo Vap tube to go dry

19. Allow the sample to cool for 10 minutes.

a. If the sample does not require GPC and a steam bath concentration or Rapid – Vap evaporation is required:

(1) Use a graduated cylinder to add approximately 50 mL of hexane directly to the K–D through the Snyder column for 1 Liter analyses or directly to the Rapid-Vap Evaporator Tube for 250 mL analyses.

(2) For 1 Liter Analyses: Concentrate until a volume of 3 to 5 mL is achieved. For 250 mL Analyses: Concentrate to around 1 mL.

(3) Remove the sample from the bath (or Rapid-Vap Evaporator) and allow the sample to cool for 10 minutes.

(4) For 1 Liter Analyses: Remove the ampule and use a wash bottle to adjust the final volume to exactly 10.0 mL with hexane in a calibrated ampule. For 250 mL Analyses: Bring to final volume 2mL in the Class A Evaporator Tube by rinsing with hexane.

Exception:

(a) If samples are scheduled for Prep 12026, remove the ampule and place on an N-evap until the volume is below 5 mL. Adjust the final volume to 5 mL with hexane in a calibrated ampule.

(b) If samples are scheduled for Prep 13093,, remove the ampule and place on an N-evap until the volume is below 2mL. Adjust the final volume to 2mL with Hexane in a calibrated ampule.

(5) Mix extract thoroughly with a disposable pipette.

b. if the sample does not require GPC and a Turbo Vap concentration is required.

(1) Use a graduated cylinder to add approximately 50ml of hexane directly to the Turbo Vap tube.

(2) Concentrate until a volume of <2 ml is achieved.

(3) Remove the sample from the Turbo Vap. Quantitatively transfer the extract with hexane into a calibrated ampule and bring to a final of 10ml for 1liter preps and to 2ml for 250ml preps.

(4) Mix thoroughly with a disposable pipette.

c. If the sample requires GPC and a steam bath concentration or Rapid-Vap Evaporation is required.

(1) For 1 Liter Analyses: Remove the ampule and adjust the final volume to exactly 10.0 mL with methylene chloride. For 250 mL Analyses: Quantitatively transfer the sample from the Evaporator Tube and bring to FV 10.0 mL with methylene chloride using a Class A volumetric.

(2) Mix thoroughly with a disposable pipette.

(3) Perform GPC cleanup following SOP 1-P-QM-PRO-9015407.

(4) Once the GPC cleanup is completed, place the extract in a K-D containing a Teflon boiling chip.

(5) Attach a 3-ball Snyder column to the K-D, wet with solvent, and concentrate the extract to approximately 3 to 5 mL on a steam bath at 85° to 99°C. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

(6) Allow the sample to cool for 10 minutes.

(7) Use a graduated cylinder to add approximately 50 mL of hexane directly to the K-D through the Snyder column.

(8) Concentrate until a volume of 3 to 5 mL is achieved.

(9) Remove the sample from the bath and allow the sample to cool for 10 minutes.

(10) Remove the ampule and use a wash bottle to adjust the final volume to exactly 5.0 mL with hexane.

(11) Mix thoroughly with a disposable pipette.

d. If the sample requires GPC and Turbo Vap concentration is required.

(1) Remove the Turbo Vap tube from the Turbo Vap. Quantitatively transfer the extract into a calibrated ampule and bring to a final volume of 10ml with methylene chloride.

(2) Mix thoroughly with a disposable pipette.

(3) Perform GPC cleanup following SOP 1-P-QM-PRO-9015407.

(4) Once the GPC cleanup is completed, place the extract in a Turbo Vap tube.

(5) Place the Turbo Vap tube in a Turbo Vap with the temperature set between 45° to 50° C. Concentrate the extract to approximately 3 to 5ml.

(6) Use a graduated cylinder to add approximately 50ml of hexane directly into the Turbo Vap tube and concentrate to approximately 2ml.

(7) Remove the sample from the Turbo Vap. Quantitatively transfer the extract into a calibrated ampule using hexane to bring to a final volume of 5ml.

20. Complete Procedure Steps 21 through 24 as needed.

21. If the extract is scheduled for analysis 6030, 10227, 12013, 14169, 14184, 14186, 14188 treat the extract with sulfuric acid as described in SOP 1-P-QM-PRO-9015477.

22. If the extract is scheduled for analysis 0177, 7572, 10589, 13634, 14134, 14166 florisil the sample 2 mL to 2 mL as described in the Pesticide Florisil section of SOP 1-P-QM-PRO-9015477.

Pesticide florisil is also being performed for analysis 0950 and 2257 if required due to matrix or client request.

23. If the extract is scheduled for analysis 6030, 10227, 12013, 14169, 14184, 14186, 14188 florisil the extract following the PCB Florisil section of SOP 1-P-QM-PRO-9015477.

24. If the extract has a sulfur odor, perform copper cleanup of the extract as described in SOP 1-P-QM-PRO-9015477 for analysis 6030 only. Other samples are cleaned up with GPC.

Calculations

See analysis method as listed in the Cross Reference section.

Statistical Information/Method Performance

See analysis method as listed in the Cross Reference section.

Quality Assurance/Quality Control

A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. Exception: For analyses referencing Methods 608 or 622 (i.e. 7572, 6030, 11119, 11123,13634, 14188) batches cannot exceed 10 field samples.

For each batch of samples extracted a blank, an LCS, an MS and an MSD must be extracted. For method 608, the laboratory must, on an ongoing basis, spike at least 10% of the samples, if enough sample volume is received, to assess accuracy. If there is limited sample preventing the preparation of the MS/MSD, an LCSD must be prepared instead. If the batch contains only field or equipment blank samples, the LCS/LCSD QC pairing must be used.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed instead.

End of document

Version history

Version	Approval	Revision information
19	07.OCT.2016	

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eurofins	Always check on-line for validity **Pesticides in Water by Method 8081B using GC-ECD**	Level: **Work Instruction**
Document number: **T-PEST-WI9999**		
Old Reference: **1-P-QM-WI-9015107**		
Version: **5**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **20-OCT-2015**	Document users: **6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C**	Responsible: **5_EUUSLA_Pesticide Residue Analysis_Manager**

LIMS ID

Analysis DOD - 10589, 10647

Revision Log

Revision: 5		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Scope	Reflects current compound list and LOQs	Updated compound list and LOQs
Statistical Information/Method Performance	Clarification	Added information on performing MDL studies

Revision: 4		**Effective Date:** Aug 7, 2014
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Clarification	Rearranged words. Pesticides in Water by Method 8081B using GC-ECD
Historical/Local Document Number	Correction	Added 10647.
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Cross Reference	Referenced in document	Added 1-P-QM-QMA-9015390.
Scope	Reflects current LOQs	Updated LOQs.
Personnel Training and Qualifications	Clarification	Added information on IDOC and DOCs
Sample Collection, Preservation, and Handling	Reflects changes in many industry standards	Updated refrigeration of samples from 4° ± 2°C to 0° to 6°C, not frozen.
Reagents and standards	Enhancement	Removed table and added information about standard database.
Calibration	Reflects current sequence setup	Added ICVs for Mix E, chlordane, and toxaphene
Calculations	Unnecessary information Correction	Removed reference to specific volumes. Changed reference from response factor to calibration factor throughout the section.
Quality Assurance/ Quality Control	Clarification	Updated to same verbiage as the other Dept 24 analysis SOPs

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8081B, Rev. 2, February 2007.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187	Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater
Analysis #6030,10227	Polychlorinated Biphenyls (PCBs) by Method 608 or 8082 in Waters
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015493	QC Data Acceptability and Corrective Action

Document	Document Title
1-P-QM-PRO-9015494	Interpretation of Chromatographic Data
1-P-QM-PRO-9015495	Preventative and Corrective GC Maintenance
1-P-QM-PRO-9015496	Monitoring QC Data Acceptance Limits
1-P-QM-PRO-9015498	Setting Up Single Component Initial Calibrations
1-P-QM-PRO-9015499	Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
1-P-QM-PRO-9015501	Common Equations Used During Chromatographic Analyses
1-P-QM-QMA-9015390	Demonstrations of Capability
1-P-QM-QMA-9017309	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is used for identifying and quantitating the following Pesticides in aqueous samples:

Compound	LOQ (µg/L)
alpha-BHC	0.01
beta-BHC	0.01
delta-BHC	0.01
gamma-BHC (lindane)	0.01
heptachlor	0.01
Aldrin	0.01
heptachlor epoxide	0.01
endosulfan I	0.01
endosulfan II	0.03
endosulfan sulfate	0.02
Dieldrin	0.02
Endrin	0.02
4,4'-DDE (*p,p*)	0.02
2,4'-DDE (*o,p*)	0.02
4,4'-DDD (*p,p*)	0.02
2,4'-DDD (*o,p*)	0.02
4,4'-DDT (*p,p*)	0.02
2,4'-DDT (*o,p*)	0.02
endrin aldehyde	0.1
endrin ketone	0.02
methoxychlor	0.1
technical chlordane	0.5
alpha-chlordane	0.01
gamma-chlordane	0.02
toxaphene	1
Kepone	0.2
Mirex	0.05

Compound	LOQ (µg/L)
Telodrin	0.01
hexachlorobenzene (HCB)	0.01
Total Endosulfan (I + II)	.01
Total DDTs	0.02

Limit of Quantitations (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

HCB, Telodrin, and Kepone, are additional compounds offered by special request.

Basic Principles

A portion of sample is extracted serially with methylene chloride. The volume of sample can be adjusted depending on the physical appearance of the sample and the amount sent for analysis. The extract is dried, concentrated, and exchanged to hexane. The pesticides are then identified and quantitated using gas chromatography with electron capture detectors (GC-ECD). Florisil, GPC, or copper cleanups may be employed to reduce matrix interferences that introduce large, unresolvable peaks into the chromatogram.

Reference Modifications

Gas Chromatography conditions differ from those listed in 8081B. However, all quality control (QC) criteria are met.

Definitions

1. Analytical Batch – A group of field and Quality Control (QC) samples of the same matrix, extracted together under the same conditions and period of time, using the same lot(s) of chemicals. The batch is limited in size to 20 field samples plus QC for SW-846 series methods.

2. Breakdown check – Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to verify the inertness of the injection port since DDT and Endrin are easily degraded in the injection port.

3. Continuing calibration verification (CCV) – A mid-level standard used to verify that the analytical response is reliable, and has not changed significantly from the current Initial Calibration curve (ICAL). The verification of the ICAL that is required during the course of analyses at periodic intervals.

4. Initial Calibration Verification (ICV) - Second source calibration verification. A standard obtained or prepared from a source independent of the source of standards for the ICAL. Used to verify the integrity of the standards used for initial calibration.

5 .Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD) – A sample of known composition analyzed with each batch of samples to demonstrate laboratory accuracy. The samples either naturally contain the analytes of interest or are clean samples fortified with known concentrations. Used to demonstrate laboratory accuracy. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

6. Matrix spike/matrix spike duplicate (MS/MSD) – A sample created by fortifying a second aliquot of a water or soil sample with some or all of the analytes of interest. The concentration added is known and compared to the amount recovered to determine percent recovery. Matrix spike recoveries provide information about the accuracy of the method in light of the matrix analyzed. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

7. Method blanks – A designated sample designed to monitor for sample contamination during the analysis process. A volume of deionized laboratory water is typically used to monitor water sample analysis, while solids blanks consist of a purified solid matrix or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

8. Surrogates – Organic compounds which are similar to the analytes of interest but are not naturally occurring in environmental samples. Surrogates are spiked into all standards and every field and QC sample prior to extraction and analysis to provide information regarding the effects of the sample matrix.

Interferences

A. Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

B. Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

C. An electron capture detector is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.

D. Extracts may require further cleanup if interferents are present. Refer to (1-P-QM-PRO-9015477) SOP-OE-004 for details on each cleanup procedure. Interfering materials can introduce large, unresolvable peaks into the chromatogram.

1. Use Florisil cleanup to reduce organics that can interfere (polar compounds).

2. Use GPC to remove sulfur and higher molecular weight organics.

3. Use copper cleanup to remove elemental sulfur.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Lab coats and safety glasses must be worn around the GC where solvents and sample extracts are handled.

All GC vials and vials containing extracts are placed in a hazardous waste container for lab pack disposal. There is a satellite container in the laboratory, which is then emptied into the main laboratory waste collection drums. All solvent waste is disposed of in solvent waste containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs. Refer to 1-P-QM-QMA-9015390 for more guidance on these options.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass containers with Teflon™-lined caps, preserved with 0.008% sodium thiosulfate in case residual chlorine is present, and stored under refrigeration at 0º to 6°C, not

frozen. Sample extraction must be performed within 7 days of collection, and sample analysis must be performed with 40 days of extraction. Extracts are stored frozen at -10º to -15ºC.

Apparatus and Equipment

1. HP 6890 gas chromatograph equipped with dual electron capture detectors, or equivalent

2. Columns:

   a. Phenomenex MR1 30 m × 0.32 mm × 0.5 µm or equivalent

   b. Phenomenex MR2 30 m × 0.32 mm × 0.25 µm or equivalent

3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.

4. Various sizes of Class−A volumetric pipettes, flasks, and syringes

Reagents and Standards

A. Reagents

1. Hexane for autosampler rinse vials, stored at room temperature.

2. UPC (ultra pure carrier) helium for carrier gas

3. UPC nitrogen for detector make-up gas

4. UPC hydrogen for carrier gas, bottled or from a generator

B. Standards

1. All standards are prepared using Class−A volumetric pipettes, flasks, and syringes.

2. All weights are made on an analytical balance.

3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

4. All prepared standard solutions are stored at −10º to -15°C in labeled containers.

5. See the electronic standards database for the compound list and concentrations contained in the various mixes.

6. PCB standards are identical to those outlined in the department Analysis #6030, 10227.

7. Mix A – Restek Catalog #32292. Equivalent to the CLP (SOW-OLMO3.2) Mix A and B, contains all single component pesticides and surrogates in the TCL and PPL organochlorine lists.

8. Mix E – Restek Custom Mix #55992. Contains additional organochlorine pesticides such as kepone, the *o,p* isomers of DDT, DDD, DDE, telodrin, mirex, and hexachlorobenzene.

9. Toxaphene stock – Restek Catalog #32005 at 1,000,000 ppb. Prepare an intermediate by placing 2 mL into a 25-mL volumetric and bring to volume with hexane.

10. Chlordane stock (CAS# 57-74-9)– Ultra Scientific PP-150 (100,000 ppb in methanol).

11. Surrogate stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetra-chloro-meta-xylene (TCX) at 200,000 ppb each in acetone.

12. Pest MS stock – Supelco Cat. #48796 and #48196. Contains all single component pesticides and surrogates in the TCL and PPL organochlorine lists.

13. EVAL stock – Restek Cat. #32074-510. Equivalent to CLP performance evaluation mix (PEM).

14. ICV stocks – These must be from a second source different than those used for working calibration standards.

Restek #32297 and #32298 for Mix A; Accustandard Cat. # S-5833 for Mix E; Supelco Cat #4-8103 for Toxaphene; Accustandard #P017S-10x for Chlordane.

15. Instrument Blank (IBLK) – surrogate stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 200,000 ppb each in acetone.

16. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (ie working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. MIXA1), parent solution name, aliquot used, final volume, solvent used, concentration or each compound in the solution, and the expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

d. See below for the preparation of the matrix spike and the surrogate spike solution used during the extraction process.

Standard Name	Parent Solution	Aliquot (mL)	Final Vol. (mL)	Solvent	Description	Expiration Date
SW-846 SS	SS Stock	1.5	1000	acetone or methanol	SW-846 Water Surrogate - identical to that prepared for PCB analyses	6 months
SW-846 MS	MS Stock	1.0	500	acetone or methanol	SW-846 Water Spike for single component organochlorines in the TCL/PPL list	6 months

17. Standards for the PCBs are run during this analysis since some of the PCB peaks may coelute or overlap with the pesticide peaks of interest. The information is used for proper identification and interpretation of the peaks observed for each sample. Quantitation of the same peak as a pesticide and PCB can be avoided.

Extraction Procedure

See Analysis #6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187.

Gas Chromatographic Conditions

Detector: ECD

Detector Temp: 330°C

Oven Temp: 140°C, no hold, 25°C/min to 250°C, then 22C/min to 300 C. hold 3 min

Carrier: Hydrogen at constant flow of 3.4 ml/min

Helium may also be substituted

Makeup Gas: N_2 at 55 mL/min

Injection Size: 1 µL, direct injection

Injector Temp: 225°C

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity, and/or improve the separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

Calibration

A. The pesticide analysis is run as a dual column approach. One injection is split onto two analytical columns. All initial and continuing calibration criteria listed below applies to both analytical columns.

B. Prior to starting a new calibration, check injection count. Change the septum on the GC every 200 injections, or more frequently if needed, and allow the system to stabilize.

C. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.

D. Prepare a sequence using the following order of injections:

1. Conditioner
2. IBLK (instrument blank)
3. EVALX
4. MIXA1
5. MIXA2
6. MIXA3
7. MIXA4
8. MIX A5
9. MIXE1*
10. MIXE2*
11. MIXE3*
12. MIXE4*
13. MIXE5*
14. TOXA1^{+} (if one level is run use TOXA1; if 5 levels are run use TOXA1 – TOXA5)
15. CHLD3^{+} (if one level is run use CHLD1; if 5 levels are run use CHLD – CHLD5)
16. AR163
17. AR2154
18. AR483
19. MDLA (MDL std for Mix A)
20. MDLE (MDL std for Mix E)
21. MDTX (MDL std for toxaphene, only if 5 level TOX curve is run)
22. MDCH (MDL std for chlordane, only if 5 level CHLD curve is run)
23. ICMAX (ICV)
24. ICMEX (ICV; if needed)
25. ICTX (I; if needed)
26. ICCH (ICV; if needed))
27. Blank
28. LCS
29. 1234567
30. 1234567ms
31. 1234567msd
32.– 42. Continue running samples for a 12-hour period from last standard
43. EVALX
44. MIXA3 (CCV)

45. MIXE3* (CCV)

E. Run a breakdown evaluation standard (EVAL) at the start of an ICAL to ensure the breakdown of DDT and endrin meets the method acceptance criteria.

1. Evaluates the system for the breakdown of DDT and Endrin before sample analysis can begin.

2. Breakdown check is run ongoing throughout the run after every 12 hours.

3. Sample analysis cannot be performed if the breakdown exceeds 15% for either compound.

F. *MIX E is only needed when analyzing for additional pesticides including 2,4−DDD, 2,4-DDE, 2,4-DDT, keptone, mirex, HCB, and telodrin.

G. The order of injections for the multi−components is not critical as long as they are all run before sample analysis.

1. Aroclors are only used to identify possible PCB patterns which may interfere with the pesticide detections.

2. A single level of toxaphene and chlordane can be run for use in identifying the presence of these target compounds; however,

a. If either of these compounds is detected in a sample, the sample must be rerun with a full five−point curve.

b. The full curve can be run at the outset of a new calibration if the samples to be run are known to contain toxaphene or chlordane.

H. The conditioner injection is usually a standard or sample that has already been injected.

1. The conditioner is used to prime the system.

2. It is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

I. Hexane blanks may also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

J. Instrument blanks (IBLK) may also be run with the continuing check standards - this is optional but frequently requested for projects.

1. The instrument blank (IBLK) is injected after the conditioners but before the initial calibration.

2. It is used to determine that the instrument is free of background noise or contamination.

K. Initial Calibration (ICAL)

1. Calibrate using the 5 levels of the single component pesticides contained in MIX A and E, and using the single point for chlordane, toxaphene (or a full curve when sample(s) contain toxaphene or chlordane).

2. An external standard calibration is used with average response factor (AVGRF) for all analytes where the % RSD is ≤20%.

3. If the RSD is > 20%, use a calibration curve.

a. Attempt a linear fit first. Use this fit if the correlation coefficient is ≥0.99.

b. If the correlation coefficient is less than 0.99, a quadratic fit will be tried.

(1) A six-point calibration must be run to use quadratic.

(2) Prepare a sixth point somewhere within the established calibration range listed in the standards preparation section.

(3) Typically the pesticides in this method will not require a quadratic fit.

(4) Quadratic fit cannot be used to extend the calibration range or bypass instrument maintenance

NOTE: For samples from South Carolina, a quadratic fit may not be used. Additionally, use of the average of the %RSDs (grand mean) for using the AVGRF as the calibration fit is not permitted.

c. For either curve type, extrapolate or force to zero is not allowed. Set the zero to ignore. See 1-P-QM-PRO-9015498 (SOP-PP-031) for more details.

d. If toxaphene or chlordane is detected in a sample, the sample will be rerun along with a full five-point calibration for that analyte as well as check standards.

e. If the 0.99 curve coefficient cannot be met:

(1) Inspect the data points to see if one or more calibration levels appear to be off.

(2) Reinject or remake the standard if a specific calibration level has concentrated due to solvent evaporation, or degraded over time.

(3) Perform instrument maintenance as needed. See 1-P-QM-PRO-9015495 (SOP-PP-013) for troubleshooting linearity problems.

f. Curve types and criteria can be altered to meet client or project specific requirements as well as any regulatory agency requirements that may differ from those listed here.

4. Set up the aroclor data in the custom “datalog” program.

a. The retention times of the peaks used for identifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations.

b. This calibration is only used to help identify potential aroclor patterns and peaks that may overlap or interfere with target pesticides.

5. Set up the toxaphene and chlordane calibration data in the custom “datalog” program.

a. The retention times of the peaks used for identifying and quantifying these multi-component pesticides are entered into the calibration file along with the corresponding peak heights and concentrations.

b. The calibration is external standard using the AVGCF.

c. Toxaphene and chlordane must meet the 20% RSD criteria.

d. See 1-P-QM-PRO-9015499 (SOP-PP-032) for details on this program.

6. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections that contain them.

7. Set the scaling of chromatograms and peak integration parameters so that the peaks for each compound of interest are detected and integrated at the concentration of the MDL. This ensures that the LOQ and MDL can be met.

L. Initial Calibration Verification (ICV)

1. Verify the calibration curve using the ICV mixtures injected directly after the full ICAL.

2. The % difference of the concentrations for these must be within 20% difference of the nominal concentration.

M. Continuing Calibration Verification (CCV)

1. Analyze a set of check standards after each set of injections in a 12-hour period, or 20 samples, whichever comes first (samples, QC, blanks, etc.).

a. Use a mid level single component standard to evaluate the response of single component analytes.

b. Run the breakdown evaluation mix (EVAL). Analysis cannot proceed if either compound exceeds 15%.

2. The concentration quantitated for the continuing calibration check standards must be within 20% difference (%D) of the nominal concentration.

3. Samples must be bracketed by compliant standards.

4. When confirmation of target analytes is needed, the initial calibration criteria must be met and the second column must meet the 20% continuing calibration criteria.

5. When a CCV fails to meet the QC criteria, all samples that were injected after the last CCV that passed must be re-injected.

Exception: If the CCV fails high and those targets are not detected in the associated samples, the samples can be reported.

6. The instrument blank (IBLK) may be injected after each set of continuing calibration verifications.

a. This is optional but frequently requested for projects.

b. It must be evaluated as a water matrix against the water MDL/LOQs.

c. The IBLK must not have any target compounds above the reporting limits.

(1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be reinjected after another compliant IBLK.

(3) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up the instrument.

7. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours, or at no less than .02 minutes, applied to the initial calibration standard, usually Level 3.

b. If the RTs for a continuing calibration standard fall outside the RT window, update the midpoint RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24−hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Make injections via an auto sampler.

2. Samples are analyzed according to the sequence in the calibration section above.

3. Retention times of peaks in the samples are compared to the standard RT windows. Peaks present on both columns (and that are also in the correct ratios to represent an aroclor) are quantitated and the high value is reported unless there are chromatographic anomalies. See 1−P−QM−PRO−9015494 SOP−PP−011).

4. Continue running groups of samples/injections followed by check standards every 12 hours or every 20 injections, whichever comes first.

5. If significant interference is present, schedule florisil cleanup. If elemental sulfur is present, copper cleanup the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract, analyze, and adjust the LOQs accordingly.

6. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

A. See 1−P−QM−PRO−9015501 (SOP−PP−040) for details on all calculations/equations used to evaluate the initial and continuing calibration.

B. Calculation of results is performed according to the following procedures:

1. Single-component compounds

   a. Using AVGCF from initial calibration

$$\frac{Sample\ Height}{AVGCF} \times \frac{FV}{IV} \times DF = \mu g/L \quad as\ received$$

b. Using linear curve from initial calibration:

$$[(Sample\ Height - Y - intercept)/Slope] \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

Where:

FV (final volume) = volume in mLs

IV (initial volume) = volume in mLs

DF (dilution factor) = as needed

2. Multi-component compounds

The peak heights generated by the integration system are used to calculate the Calibration Factors (CF) for peaks of interest for each quantitation peak used for toxaphene and chlordane.

Usually the six major peaks that are unique toxaphene and chlordane (include alph chlordane, gamma chlordane and heptachlor) are chosen for quantitation. Sample concentrations are calculated per peak using Average Calibration Factor (AVGCF).

$$\frac{Sample\ Height}{AVG\ CF\ (CF)} \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

Where:

FV (final volume) = volume in mLs

IV (initial volume) = volume in mLs

DF (dilution factor) = as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(Result\ 1 + Result\ 2 + \ldots + Result\ n) / n = Average\ Result$$

3. A breakdown mix (EVALX) containing p,p′-DDT and Endrin is run to check for breakdown. The breakdown must not exceed 15% for either compound. Breakdown is calculated as:

$$\%\ Breakdown\ for\ p,p'\text{-}DDT = \frac{pk\ ht\ (area)\ of\ p,p'\text{-}DDE + p,p'\text{-}DDD}{pk\ ht\ (area)\ of\ p,p'\text{-}DDE + p,p'\text{-}DDD + p,p'\text{-}DDT} \times 100$$

$$\%\ Breakdown\ for\ Endrin = \frac{pk\ ht\ (area)\ of\ Endrin\ Aldehyde + Endrin\ Ketone}{pk\ ht\ (area)\ of\ Endrin\ Aldehyde + Endrin\ Ketone + Endrin} \times 100$$

If breakdown fails, injector maintenance must be performed. Analysis cannot proceed until breakdown check passes.

Statistical Information/Method Performance

Generate method detection limits (MDLs) and limits of quantitation (LOQs) according to (1−P−QM−QMA−9017309) LOM−SOP−ES−203. Initially, perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire extraction and analysis procedure. Compare and pool results to determine the final reporting MDL. An MDL study or verification of the MDL is required each year. NELAC allows for an annual verification of the MDL in lieu of a full MDL study. The department management maintains annual study data. Updates to the LIMS are made as needed by the QA department and only as directed by the department manager. Update the department database via a download from the LIMS.

QC limits for surrogates, LCS/LCSD, and MS/MSD are established through statistical analysis of historical data. The limits are evaluated every 6 months and updated as needed. The limits are maintained in the LIMS for the relevant analysis numbers. See 1−P−QM−PRO−9015496 (SOP−PP−025) for further information on monitoring and establishing limits.

Quality Assurance/Quality Control

Each extraction batch (up to 20 samples) must contain a method blank, a laboratory control spike sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD) or an LCS/LCSD.

The TCL single-component pesticides of interest for each analysis are routinely spiked. Mirex, o,p−DDE/DDD/DDT, telodrin, and HCB are not spiked since this would result in co−elution with the other

spiked compounds. These can be spiked at a client's request for special projects, within our scope of accreditation.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

See 1-P-QM-PRO-9015493 (SOP-PP-002) for details on QC acceptance criteria and corrective action.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

End of document

Version history

Version	Approval	Revision information
5	20.OCT.2015	

eurofins	Always check on-line for validity **Microwave Extraction by Method 3546 for Semivolatiles**	Level: **Work Instruction**
Document number: **T-OE-SVOA-WI10928**		
Old Reference: **1-P-QM-WI -9015105**		
Version: **12**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **14-JUN-2017**	Document users: **5_EUUSLA_GC/MS Semivolatiles_Manager, 5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Semivolatile Soils, 6_EUUSLA_GC/MS Semivolatiles_Analysts**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis DOD - 10498, 10487, 10809, 10810, 10811, 10812, 10813, 10814, 11630, 11916

Revision Log

Revision 12		**Effective Date:** This version
Section	**Justification**	**Changes**
LIMS ID	Reflects current LIMS scan numbers	Added Analysis #10487
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout Document	Reflects current document numbers	Removed old document numbers and replaced with D4 document numbers
Procedure 6	Reflects current practice	Added spikes for Analysis #6397

Revision: 11		**Effective Date:** October 4, 2016
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Reagents and Standards 4.	Requirement for SC	Added Ottawa Sand
Procedure 4.	Requirement for SC	Added Ottawa Sand for QC for SC requirement

Reference

1. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 3546, Revision 0, February 2007.

2. *MARS Operation Manual*, Revision 2, February 2006.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0949, 1309, 1476, 1536, 1946, 1947, 1953, 2035, 2395, 4615, 4678, 4688, 6387, 6397, 7804, 7805, 10032, 10723, 10724, 10727, 10728, 13615, 13618	Semivolatile Organic Compounds, Including DRO/ORO, by Method 8270C in Aqueous and Non-Aqueous Matrices Using GC-MS
Analysis #2487	Food and Tissue Preparation
Analysis #4220, 4221	Determination of Tetraethyl Lead (TEL) and Tetramethyl Lead (TML) by GC/MS Analysis
Analysis #8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971	Semivolatiles by Methods 8270C/D SIM
Analysis 10242, 10262, 11305, 11597	Determination of Parent and Alkyl Substituted Polynuclear Aromatic Hydrocarbons (PAHs), Alkanes and Geochemical Biomarkers by Gas Chromatography/Mass Spectrometry (GC/MS-SIM)
Analysis #10461, 10462, 10726	Semivolatile Organic Compounds by Method 8270D in Aqueous and Non-Aqueous Matrices using GC-MS
Analysis #10962, 11622	Determination of N-Nitrosodimethylamine (NDMA) in Water and Soil by EPA 1625C
T-OE-SVOA-WI10554	Semivolatile Extract Cleanup Using Gel Permeation Chromatography
T-SVOA-WI11998	Semivolatile Spiking and Calibration Standards
T-OE-GEN-WI10864	Glassware Cleaning for Organic Extractions
T-OE-GEN-WI10876	Organic Extraction Standards Storage and Handling

Scope
This procedure is used to clearly outline the steps taken for the extraction of semivolatile compounds from soils or solid wastes using microwave technology.

Basic Principles
A portion of sample is placed in an extraction vessel. Surrogate standards are added to each sample to monitor recovery. The vessel is then loaded into the instrument and extracted. The organic compounds present in the sample dissolve in the solvent, which is then removed. The extract is concentrated to about 1 mL. At this time, it is determined if the sample requires gel-permeation cleanup (GPC). If needed, the extract is diluted to 10 mL, then cleaned using GPC and concentrated to 0.5 mL. If GPC is not necessary, the extract is brought to 1.0 mL and bottled. The extract is stored in an amber-autosampler vial in the freezer until analysis.

Reference Modifications
Base/Neutral and Acid compounds are added at a concentration of 50 ppm in the matrix spiking and LCS solutions so that the concentrations in the extract are within calibration range.

After Procedure Step 24, the joint of the K-D is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

Interferences
Impurities in solvents, reagents, glassware, or other hardware used in sample processing can interfere with the method. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling
All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are then transferred to the lab-wide disposal facility. Any solid waste

material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meet the defined acceptance criteria. The criteria for the LCSs include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation and Handling

Samples are collected in glass wide-mouth jars with PTFE-lined lids and stored under refrigeration at 0° to 6° C, not frozen, prior to extraction. Samples must be extracted within 14 days of sample collection. Extracts are stored in the freezer at ≤-10° C and must be analyzed within 40 days of the date extracted.

Apparatus and Equipment

1. MARS Xpress – CEM Corp. or equivalent

2. Kuderna-Danish (K-D) assembly with appropriate ampule for concentrating the solvent used during the extraction

3. Steam bath, VWR/LLI Model #1127 or equivalent

4. Balance – Capable of weighing to 0.01 g

5. Beakers – Stainless steel – Assorted sizes

6. Scoop

7. Pipettes – Class A, assorted sizes

8. Wash bottles – Teflon®

9. Teflon®-boiling chips

10. Micro Snyder columns

11. Disposable pipettes

12. Autosampler vials – Amber, crimp top

13. Funnels – Stainless steel or Teflon®

14. Extraction vessels

15. Frits – Various

16. Sodium sulfate columns

Reagents and Standards

1. Methylene chloride (CH_2Cl_2) – Pesticide grade or equivalent. Store at room temperature for up to one year.

2. Acetone – Pesticide grade or equivalent. Store at room temperature for up to one year.

3. Sodium Sulfate (Na_2SO_4) – Reagent grade or equivalent. Bake at 400°C for a minimum of 4 hours in a shallow pan to remove contaminants. Store in a glass jar for up to 1 year after baking.

4. Ottawa sand - Bake at ~ 400° C for a minimum of 4 hours to remove organic contaminants. Store in a glass jar for up to 1 year after baking.

NOTE: All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical analyses for scans 4221, 10242, 10723, 10724, 10725, 10726, 11622 and 11917.

Calibration

Not applicable to this procedure.

Preparation of Glassware

See *T-OE-GEN-WI10864*.

Procedure

1. Sample aliquot

a. If Sample Registration has pre-weighed the sample into a glass jar, add 5g of sodium sulfate, mix and proceed.

b. If the sample is not pre-weighed, weigh 30.0 - 30.5 g of sample into a labeled stainless steel beaker.

(1) Record the initial weight and any comments about the sample in the extraction log.

(2) Alternative sample weights may be used to meet certain reporting limits. However, if sample weight <30.5 is used the sample must be divided among multiple vessels and the extracts combined.

c. Process all tissues by *Analysis #2487* prior to extraction.

2. Add 5 g of sodium sulfate to each sample and mix.

a. If the sample has high water content or is a clay-like soil, add an additional 10 g of sodium sulfate.

b. Mix the sodium sulfate and sample until a free-flowing consistency is reached.

3. Perform the background, matrix spike (MS), and matrix spike duplicate (MSD) on three separate aliquots of a field sample.

4. Prepare the Blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) (if applicable) by filling a Teflon® extraction vessel with 35 g of sodium sulfate.

NOTE: For samples from SC, use Ottawa Sand for the preparation of the Blank, LCS, and LCSD (if applicable).

Record 30.0 g on the extraction log. (The sodium sulfate used for the QC samples is measured out as 35.0 g to account for the 30 g "sample" plus the 5g of sodium sulfate.)

5. Place each sample into its clearly marked corresponding extraction vessel. Use a funnel to prevent spillage and loss of sample.

6. Add surrogate standards and spiking solutions using pipettes. Add surrogates to all samples, blanks and spiked samples. Add spikes to the LCS, LCSD (if applicable), MS, and MSD samples. Typically they are as follows:

a. Analysis 10727 – Surrogate: 1.0mL of BNA surrogate. Spike: 1.0mL of LCS spike.

b. Analysis 10725 – Surrogate: 1.0mL of BNA surrogate. Spike: 1.0mL of SIM LCS spike.

c. Analysis 10728 – Surrogate: 1.0mL of BNA surrogate. Spike: 1.0mL of LCS Spike. If required, spike 1.0 mL of Benzenthiol spike in a separate LCS/LCSD.

d. Analysis 10723, 10726, 1947 – Surrogate: 1.0mL of BNA surrogate. Spike: 1.0mL each of LCS and AppIX Mix #1. If required, Spike 1.0 mL of AppIX Mix #2 in a separate LCS/LCSD.

e. Analysis 10724 – Surrogate: 1.0mL of BNA Surrogate. Spike: 1.0mL of LCS spike.

f. Analysis 4221 – Surrogate: 1.0mL of BNA Surrogate. Spike: 1.0mL of TEL/TML spike.

g. Analysis 10242 – Surrogate: 1.0 mL of BNA surrogate. Spike: 1.0 mL of "Client Specified" spike. If required, 1.0 mL of DPnB spike.

h. Analysis 11622 – Surrogate: 1.0 mL of NDMA Surrogate. Spike: 1.0 mL of NDMA spike

i. Analysis 11917 – Surrogate: 1.0 mL of BNA surrogate. Spike: 1.0mL of SIM LCS.

j. Analysis 06397 - Surrogate: 1.0 mL of BNA surrogate. Spike: 1.0 mL of LCS matrix spiking solution and 1.0 mL of the Appendix IX Mix #1 spike is added. If required, prep a separate LCS/LCSD with 1.0 mL of Appendix IX Mix #2

NOTE: If the sample is being analyzed for Benzoic Acid, a separate LCS/LCSD must be created with 1.0 mL of Appendix IX Mix#1 spike.

See *T-OE-GEN-WI10876* for storage and handling of spikes and *T-SVOA-WI11998* for SVOA spiking and calibration standards.

NOTE: If the sample is being divided among multiple vessels, the spike must be divided among the vessels as well.

7. Add 30 mL of 50% acetone in methylene chloride to each vessel.

8. Cap each vessel according to manufacturer's guidelines.

9. Invert each vessel to ensure mixing of sample and solvent.

10. Place the vessels into the carousel. When all samples are loaded, place the carousel into the microwave.

11. Run Program "LL Soils". See Table I for Instrument conditions. Verify that the run reaches the 100 °C temperature requirement and document on batchlog.

12. Uncap the cooled vessel.

13. Pour the extract and sample into a column filled with approximately 10cm of sodium sulfate on top of a Kuderna-Danish (K-D) assembly containing a Teflon®-boiling chip.

14. Rinse the vessel with 10 to 20 mL of 50% acetone in methylene chloride from a wash bottle.

15. Place a 3-ball Snyder column on the K-D set-up, wet the column with 50% acetone in methylene chloride.

16. Concentrate over a steam bath at 84° to 99°C until the apparent volume in the ampule reaches 1 to 2 mL. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. Adjust the vertical position of the apparatus and the water temperature as needed to complete the concentration in 10 - 20 min.

17. Allow the sample to cool 10 minutes. Approximately 3 mL condenses into the ampule during this time.

18. Attach the ampule of the K-D to a micro-Snyder column, and concentrate the extract to below 1 mL. Allow the sample to cool.

19. If GPC is not needed, go to Procedure Step 25.

a. If GPC is needed, dilute the extract to 10 mL with methylene chloride.

b. Place the extract into the appropriate GPC queuing area for storage until the cleanup is performed. See *T-OE-SVOA-WI10554*.

c. Once the GPC clean up is completed, go to Step 20.

20. Place the extract in a Kuderna-Danish containing a Teflon®-boiling chip.

21. Place a 3-ball Snyder column on the set-up, wet the column with methylene chloride.

22. Concentrate over a steam bath at 80° to 90°C until the apparent volume reaches 1 to 2 mL.

23. Allow the sample to cool at least 10 minutes. Approximately 3 mL condenses into the ampule at this time.

24. Attach the ampule of the K-D to a micro-Snyder column, and concentrate the extract to below 0.5 mL. Allow the sample to cool.

25. Bring to a final volume of 1.0 mL (0.5 mL if GPC was performed) with methylene chloride in a labeled amber GC vial.

a. The final volume is determined by placing the extract into an amber-autosampler vial and comparing the level in the vial to a reference vial containing the exact targeted final volume.

b. The vials used for bottling must be the same lot number as the reference vial.

c. Methylene chloride is added to the extract using a disposable pipette until exactly the same level is in both vials.

d. If too much solvent is added to the sample vial, remove the extract from the vial and ccentrate it by micro-snydering to slightly less than the targeted final volume and rebottle.

e. Cap the vial and store in the freezer until analysis.

f. Record the final volume in the extraction log.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
For each batch of samples extracted, a method blank, an LCS, an unspiked background sample, an MS, and an MSD must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead.

A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Table I

Instrument Conditions

Power:	1600W
Ramp Temperature:	100°C
Ramp Time:	30 minutes
Hold Time:	10 minutes
Cool Down Time:	20 minutes

T-OE-GEN-WI10864 Glassware Cleaning for Organic Extractions
T-OE-GEN-WI10876 Organic Extraction Standards Storage and Handling
T-OE-GEN-WI7154 Food and Tissue Preparation
T-OE-SVOA-WI10554 Semivolatile Extract Cleanup Using Gel Permeation Chromatography
T-SVOA-WI11998 Semivolatile Spiking and Calibration Standards
T-SVOA-WI9252 Determination of Parent and Alkyl Substituted Polynuclear Aromatic Hydrocarbons (PAHs), Alkanes and Geochemical Biomarkers by Gas Chromatography/Mass Spectrometry (GC/MS-SIM)
T-SVOA-WI9553 Determination of N-Nitrosodimethylamine (NDMA) in Water and Soil by EPA 1625C
T-SVOA-WI9587 Determination of Tetraethyl lead (TEL) and Tetramethyl lead (TML) by GC/MS Analysis
T-SVOA-WI9617 Semivolatile Organic Compounds by Method 8270D in Aqueous and Non-Aqueous Matrices using GC-MS
T-SVOA-WI9623 Semivolatile Organic Compounds, Including DRO/ORO, by Method 8270C in Aqueous and Non-Aqueous Matrices Using GC-MS
T-SVOA-WI9995 Semivolatiles by Methods 8270C/D SIM

End of document

Version history

Version	Approval	Revision information
11	04.OCT.2016	
12	31.MAY.2017	

An error has happened on file: /D4Doc/formularer/2016/09/Quality Document # 1-P-QM-WI -9015105_10_1049810809.doc
Errortext: The remote server returned an error: (404) Not Found.

eurofins	Always check on-line for validity **Semivolatiles by Methods 8270C/D SIM**	Level: **Work Instruction**
Document number: **T-SVOA-WI9995**		
Old Reference: **1-P-QM-WI-9015192**		
Version: **13**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **23-AUG-2017**	Document users: **5_EUUSLA_GC/MS Semivolatiles_All**	Responsible: **5_EUUSLA_GC/MS Semivolatiles_Manager**

LIMS ID

Analysis DOD - 8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971

Revision Log

Revision: 13	**Effective Date:** This version	
Section	**Justification**	**Changes**
Throughout Document	Reflect new document numbers	Updated document references to D4 numbers
References	Most current version	Added reference for Method 8000D
Calibration C.	Enhancement	Added % Error verification for initial calibration levels

Revision: 13	**Effective Date:** This version	
Procedure B.	Reflects current practices	Updated tuning procedure requirements
Calculations	Enhancement	Added calculation for % Error
Quality Assurance/ Quality Control	Reflect current practices	Added LLOQ information

Revision: 12	**Effective Date:** Sep 08, 2016	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Formatting requirement	Replaced temperature -10°C to -15°C with ≤ -10°C; 4°± 2°C with 0° to 6°C, not frozen
Personnel Training and Qualifications	Reflect current practice	IDOCs use the calculation of percent recoveries rather than the mean accuracy and standard deviation
Reagents and Standards 3 and 5	Reflect current practice	Update to reflect current vendor and concentration used
Calibration B,C,D	Enhancement	Moved calculations to the Calculations Section
Procedure A.1 a and b	Reflect current practice	Update to reflect current vendor used
Procedure A.2	Reflect current practice	Update to reflect current mix and concentrations used
Procedure A.3a	Clarification	Add “approximate” to the dilutions performed
Procedure A.3.c and d	Clarification	Remove reference to formatted and bound standards notebook and add reference to the SEMIS folder located on the G: drive of the network
Procedure C.2	Reflect current practice	Add Bis(2-chloroethyl)ether, Dibenzofuran, Butylbenzylphthalate, and Bis(2-ethylhexyl) phthalate to groups
Procedure C.2 Group 3	Correction	Change Diethylphthalate to Dimethylphthalate
Procedure C.2 Group 4	Reflect current practice	Remove Pentachlorophenol – no longer a compound of interest
Procedure C.2 Group 5 and 6	Reflect current practice	Remove Benzidine – no longer a compound of interest and move Butylbenzylphthalate to Group 6 to reflect current practice
Procedure F	Enhancement	Moved calculations to the Calculations Section
Quality Assurance/Quality Control	Reflect current practice	Include allowance of phthalates < LOQ
Figure 1	Reflect current practice	Add compounds to Absolute PAH Standard list
Table IV	Enhancement	Add table for internal standards and corresponding compounds

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8270D (SIM), February 2007.
2. Test Methods for Evaluating Solid Wastes, SW-846 Method 8270C (SIM), December 1996.
3. Test Methods for Evaluating Solid Wastes, SW-846 Method 8000B (SIM), December 1996.
4. Test Methods for Evaluating Solid Wastes, SW-846 Method 8000D, July 2014.

5. *Hewlett-Packard Operations Manuals.*

6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Q-EQA-FRM6869	Nonconformance Form
T-SVOA-WI9598	GC/MS Preventative and Corrective Maintenance
T-SVOA-WI19594	GC/MS Audit Process
QA-SOP11178	Demonstrations of Capability

Scope

This method is suitable for the determination of low−level semivolatile compounds (PAHs) from soils and waters by selected ion monitoring (SIM) gas chromatography/Mass Spectrometry (GC/MS). The analysis applies to a concentration range that spans from an MDL of 0.01 µg/L in water (0.33 µg/kg for soil) to an upper calibration concentration of 10 µg/L in water (330 µg/kg for soil).

Basic Principles

An environmental sample (soil, water, sludge, etc.) is solvent extracted and then analyzed by electron impact gas chromatography/mass spectrometry (EI GC/MS). By analyzing for specific masses over a narrow mass range, SIM analysis allows for longer dwell times at specific masses. This allows for significantly greater sensitivity of analysis with a small reduction in specificity of analysis.

Interferences

Sample matrices can have an effect on the ability of the GC/MS system to resolve the individual masses used for quantification. Samples may require dilutions or a reduction in the extraction volume/weight used in order to achieve proper target separation and determination.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity of each reagent has not been precisely determined. However, each reagent should be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of percent recovery. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs. Refer to QA-SOP11178 for more guidance on these options.

Sample Collection, Preservation, and Handling

Water samples are collected in glass bottles with PTFE-lined lids and stored at 0° to 6°C, not frozen, prior to extraction. Samples must be extracted within 7 days of collection. Extracts are stored in the freezer at ≤ -10°C.

Soil samples are collected in wide-mouth glass jars with PTFE-lined lids and stored at 0° to 6°C, not frozen, prior to extraction. Samples must be extracted within 14 days of collection. Extracts are stored in the freezer at ≤ -10°C.

Apparatus and Equipment

1. Hewlett-Packard Model 5890 Gas Chromatograph or equivalent

2. Hewlett-Packard Model 5971, 5972, or Agilent Model 5973 Mass Selective Detector or equivalent

3. Hewlett-Packard Chemstation Software, Thru-Put Systems Target data system or equivalent

4. Volumetric flask – Assorted sizes

5. Hamilton Gastight syringe – Assorted sizes

Reagents and Standards

1. Tune Check Solution – 50−ng/µL solution of decafluorotriphenylphosphine (DFTPP) containing pentachlorophenol, benzidine and *p,p'*−dichlorodiphenyltrichloroethane (DDT) in methylene chloride, Absolute Standards Inc. Catalog #43030, or equivalent; store at 0° to 6°C, not frozen

2. Methylene chloride – (MeCl2 or CH2Cl2) – Baker Ultra Resi–Analyzed, or equivalent

3. Internal standard solution – 2000 ppm in methylene chloride Restek Catalog #A073516, or equivalent

4. Absolute PAH Standard – 1000−ppm solution of PAHs in methylene chloride, Catalog #93462, or equivalent

5. 8270 Surrogate standard solution – 1000−ppm solution in methylene chloride, Restek Catalog #A0114172, or equivalent

6. Restek (ICV) Solution Kit – 2000 ppm in methylene chloride, or equivalent

7. Quinoline – 1000 ppm in methylene chloride, Absolute Standards Catalog #70353, or equivalent

8. Benzenethiol – 1000 ppm in methylene chloride, Absolute Standards Catalog #70900, or equivalent

9. Hexachlorobenzene – 1000 ppm in acetone, Absolute Standards Catalog, #70195, or equivalent

10. 1,2-Diphenylhydrazine – 1000 ppm in methylene chloride, Absolute Standards Catalog #70024, or equivalent

11. EPA Method 8070 – Nitrosamines - 2000 ppm in Methanol, Absolute Standards Catalog #19222 or equivalent

12. EPA Method 606 – Phthalates – 2000 ppm in Methanol, Absolute Standards Catalog # 19242 or equivalent

13. 1,4-Dioxane – 2000 ppm in methanol, Absolute Standards Catalog #90871 or equivalent

NOTE: Special compounds may be added to spikes and calibration standards as necessary. These compounds and concentrations are determined by the analyst and added in appropriate proportions to achieve proper concentrations. If a modification to a solution's final volume is required, adjust the volume of stock solutions added to achieve the appropriate concentrations.

Calibration

A. Calibration is accomplished using an internal standard calibration technique. Calibration standards at five or more concentration levels are analyzed. The relative response factor is calculated for each compound at each concentration level. The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of concentration.

1. If the performance criteria are met for the DFTPP solution (from Procedure step B.), perform an initial calibration by analyzing at least five or more calibration standard solutions referenced previously.

2. After the calibration standard analyses, inject the MDL/LOQ solution. The GC/MS system must be able to detect the compounds in the MDL/LOQ solution. If the system does not detect the compounds, then the tuning and calibration procedure must be repeated under conditions that will yield a detection for the compounds in the MDL/LOQ solution. Instrument maintenance as outlined in T-SVOA-WI9598 may be required.

3. After the MDL/LOQ solution has been run, inject the ICV solution. This standard must be analyzed after the initial calibration standards and before any samples are analyzed.

B. Calculate the relative response factor for each compound and surrogate for each calibration solution. Refer to the Calculations Section for equation.

C. For each compound and surrogate, calculate the average RRF from the RRFs of the calibration standards. For 8270D, each compound must meet the minimum response factor criteria listed in Table III. Refer to the Calculations Section for equation.

For 8270C, if the %RSD of the response factors is less than or equal to 15%, then the average relative response factor $(\overline{RRF})$ is used for quantitation. If the %RSD exceeds 15% then an alternate curve fit must be used for quantitation. A linear curve fit is a suitable alternative when only 5 calibration standards have been analyzed.

For 8270D, if the %RSD of the response factors is less than or equal to 20%, then the average relative response factor is used for quantitation. If the %RSD exceeds 20% then an alternate curve fit must be used for quantitation. A linear curve fit is a suitable alternative when only 5 calibration standards have been analyzed.

The % Error is measured between the calculated and expected amounts of an analyte at each calibration level to determine the calibration function acceptability for linear and non-linear curves. Refer to the Calculations Section for equation. The % Error should be ≤ 30% for all standards. For some data uses, ≤ 50% may be acceptable for the lowest calibration level.

D. Continuing calibrations

Verify the MS tune and initial calibration at the beginning of every 12−hour work shift during which analyses are performed.

1. The MS tune is verified following the instructions given in Procedure B. If the mass spectrum of the DFTPP peak does not meet the abundance criteria described in Table I for 8270C and Table II for 8270D, the mass spectrometer must be retuned until all criteria are met.

2. A continuing calibration verification (CCV) is analyzed after the tune each day a curve is not analyzed. For 8270D, each compound must meet the minimum response factor criteria listed in Table III. Refer to the Calculations Section for equation.

The % Drift for each compound should not be greater than ±20%. If the %Drift exceeds 20%, then corrective action must be taken and a new initial calibration may need to be performed.

3. The absolute areas of the quantitation ions of the internal standard must fall within −50% to +100% of the areas from the mid-level calibration standard produced during the last initial calibration. If the area for each internal standard is not within this window then corrective action must be taken and documented.

Procedure

The GC/MS is configured in the selected ion monitoring (SIM) mode with a total cycle time (including voltage reset time) of 1 second or less. The GC/MS system must be capable of meeting the following specifications:

Mass range:	35 to 500 amu
Scan time:	1 scan/sec
GC column:	30 m × 0.25 mm × 0.5 µm film Restek Rxi – 5 or equivalent
Injector temperature:	250° to 300°C
Transfer line temperature:	250° to 300°C
GC Oven:	Time and Temperature Programmable

EI condition:	70 eV
Mass scan:	Capable of SIM scanning (see manufacturer's instructions)
Carrier gas:	Helium at approximately 30 cm/sec.

A. Standard preparation

1. Internal standard

Internal standard mix is added to all standards and subsequent samples.

a. Soils and 1000 mL water extractions are at a concentration of 1 µg/mL. Using a 25-ul syringe, 10 µL of RestekSemivolatile Internal Standard Mix or equivalent (100 µg/mL in methylene chloride) is added to the 1 mL of standard or sample extract.

b. 250 mL water extractions are at a concentration of 0.25 µg/mL. Using a 25-µl syringe, 10 µl of RestekSemivolatile Internal Standard Mix or equivalent (25 µg/ml in methylene chloride) is added to the 1 mL of standard or sample extract.

2. Stock standard preparation

To prepare the 100−ppm and 10−ppm PAH working stocks, using a Hamilton Gastight syringe, measure the following stocks into 10−mL volumetric flasks and dilute to volume with methylene chloride. See Figure 1 for a list of compounds. Stocks are prepared every 6 months or sooner, if comparison with the QC check samples indicates a problem.

Mix	Concentration (ppm)	100−ppm Stock Amount Added (µL) to 10 mL	10−ppm Stock Amount Added (µL) to 10 mL
PAH Standard	1000	1000	100
Custom SIM Surrogate	1000	1000	100
1,2-Diphenylhydrazine	1000	1000	100
Hexachlorobenzene	1000	1000	100
Pentachlorophenol	1000	1000	100
Benzidine	10000	2000	200
Nitrosamines	2000	500	50
Phathalates	2000	500	50
1,4-Dioxane	1000	1000	100

3. Calibration levels

Standard ID	Preparation	Final Volume
Level 1: LPAH0.05	5 µL of 10−ppm working stock + 10 µL of 100−ppm internal standard	To 1.0 mL $MeCl_2$

Level 2: LPAH0.2	20 µL of 10−ppm working stock + 10 µL of 100−ppm internal standard	To 1.0 mL $MeCl_2$
Level 3: LPAH0.5	50 µL of 10−ppm working stock + 10 µL of 100−ppm internal standard	To 1.0 mL $MeCl_2$
Level 4: LPAH001	10 µL of 100−ppm working stock + 10 µL of 100−ppm internal standard	To 1.0 mL $MeCl_2$
Level 5: LPAH005	50 µL of 100−ppm working stock + 10 µL of 100−ppm internal standard	To 1.0 mL $MeCl_2$
Level 6: LPAH010	100 µL of 100−ppm working stock + 10 µL of 100−ppm internal standard	To 1.0 mL $MeCl_2$
MDL/LOQ standard: (0.01ppm)	1 µL 10−ppm working stock + 10 µL of 100−ppm internal standard	To 1.0 mL MeCl2

a. For samples extracted with miniature separatory funnels (250 mL), the calibration standards listed in the above table will be diluted approximately 4x. The calibration range will be from 0.0125 µg/ to 2.5 µg/L. The MDL standard and ICV will also be diluted 4x.

b. One of the calibration standards must be at a concentration near, but above, the limit of quantitation (LOQ). The other concentrations correspond to the expected range of concentrations found in samples or should define the working range of the GC/MS system.

c. The concentration of the MDL standard may require modification dependant upon the current MDL values. Document the procedure concentrations and volumes in the SEMIS folder located on the G: drive of the network.

d. In addition to the calibration standards, initial calibration verification (ICV) standard must be prepared using a separate source from the working stock. The concentration of the ICV standard is at or around the mid−Level of the calibration curve. A PAH spike mix different from the working stock is diluted from 2000 ppm to 1 ppm by taking 0.5 µL of the spike mix and diluting to 1.0 mL. As referenced in 1−P−QM−PRO−9015455 (SOP-EX-009) there must be an ICV standard form that compares the average relative response factors from the ICAL with the response factors from the ICV standard. The ICV recovery must be within ±30% or approval is required for those targets outside of this window. The ICV must be listed on the form 5 for the tune. The preparation of these standards is recorded in the SEMIS folder located on the G: drive of the network. The calibration standards are prepared approximately every 2 weeks, or as needed based on the frequency of analysis and contingent upon successfully passing continuing calibration check standards.

e. All standards are stored at 0° to 6°C and protected from light.

B. Tuning

1. The DFTPP tune check standard is used to assess GC column performance and injection port inertness as well as mass spectrometer performance. The GC/MS system's tune is checked by inspecting the mass spectrum of the DFTPP peak and column performance and injection port inertness

is evaluated with pentachlorophenol, benzidine, and DDT.

2. DFTPP must meet the criteria specified in Table I for 8270C and Table II for 8270D. If these criteria are met, standardization may begin at this point. If DFTPP fails the specified criteria, corrective action must be taken and the DFTPP reinjected until acceptable criteria are obtained. Tuning and DFTPP evaluation must take place every 12 hours that analyses are to be performed.

3. DDT breakdown must be <= 20% and tailing factors for benzidine and pentachlorophenol must <=2. Note that DDT breakdown greater than 20 percent may be acceptable if you are calibrating for polynuclear aromatic hydrocarbon compounds only. Consult supervisor when this situation occurs.

C. Method set-up

1. Prior to analysis by SIM (Selective Ion Monitoring) a check standard must be analyzed in the full scan mode to determine where the specific start and stop times are for each of the groups.

NOTE: Each program must be identical for the Temperature and Pressure programs so that the peak retention times are the same.

2. Each time column maintenance is performed, the scan times are reviewed to ensure that all ions are included. The instrument setup in the method is as follows. In the Set Acquisition Mode, select SIM. See specific Group for Dwell time in msec. Set the Low Res to “N” and the Start Time for each group. In the m/z field, enter the ions for each group from the following tables:

GROUP 1 (Dwell – 60 msec)			
Compound	***1.1.1.1 Type***	Quant Ion (m/z)	Monitor Ion (m/z)
1,4-Dioxane	Target	88	58
N-Nitrosodimethylamine	Target	74	42

GROUP 2 (Dwell – 40 msec)			
Compound	***1.1.1.2 Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Quinoline	Target	129	102
Benzenethiol	Target	110	66
1,4-Dichlorobenzene-d4	ISTD	152	150
Naphthalene-d8	ISTD	136	68
Naphthalene	Target	128	129
Bis(2-chloroethyl)ether	Target	93	63

GROUP 3 (Dwell – 40-50 msec)			
Compound	1.2 Type	Quant Ion (m/z)	Monitor Ion (m/z)
Dimethylphthalate	Target	163	194
2-Methylnaphthalene	Target	142	141
1-Methylnaphthalene	Target	142	141
1-Methylnaphthalene-d10	Surrogate	152	
Acenaphthylene	Target	152	76
Acenaphthene-d10	ISTD	164	162
Acenaphthene	Target	154	152

GROUP 4 (Dwell – 25-30 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Dibenzofuran	Target	168	139
Diethylphthalate	Target	149	177
Fluorene	Target	166	165
Hexachlorobenzene	Target	284	142
1,2-Diphenylhydrazine	Target	77	182
Phenanthrene-d10	ISTD	188	94
Phenanthrene	Target	178	176
Anthracene	Target	178	176

GROUP 5 (Dwell – 50 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Di-n-butylphthalate	Target	149	150
Fluoranthene	Target	202	101
Pyrene	Target	202	101
Flouranthene-d10	Surrogate	212	106

GROUP 6 (Dwell – 40 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Butylbenzylphthalate	Target	149	91
Benzo(a)anthracene	Target	228	226
Chrysene-d12	ISTD	240	236

Chrysene	Target	228	226
Bis(2-ethylhexyl)phthalate	Target	149	167
Di-n-octylphthalate	Target	149	150
Benzo(b)fluoranthene	Target	252	253
Benzo(k)fluoranthene	Target	252	253
Benzo(e)pyrene	Target	252	253
Benzo(a)pyrene	Target	252	253
Perylene-d12	ISTD	264	260
Benzo(a)pyrene-d12	Surrogate	265	132
Perylene	Target	252	253

GROUP 7 (Dwell – 100 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Indeno (1,2,3-cd) pyrene	Target	276	138
Dibenz (a,h) anthracene	Target	278	139
Benzo (g,h,I) perylene	Target	276	138

D. Analysis of Samples:

1. Analyze a predetermined aliquot of each sample extract under the same conditions used for the initial and/or continuing calibrations.

2. At the conclusion of data acquisition, use the same software to tentatively identify peaks within the retention time window of interest. Examine the ion abundances of components of the chromatogram. If the ion abundance of the main ion used for quantitation exceeds the calibration range, dilute the aliquot and reanalyze. When preparing dilutions, add sufficient internal standard to maintain the same concentration that was used.

E. Qualitative Analysis:

1. A compound is identified by comparison of the sample mass spectrum (after background subtraction) with the mass spectrum of a standard of the target compound (standard reference spectra).

2. In order to verify identification, the sample component relative retention time (RRT) must be within 10 seconds of the RRT observed for the component when a calibration solution was analyzed.

F. Quantitative Analysis:

When a compound has been identified, quantitation is based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. Refer to the Calculations Section for the equations.

Calculations

Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Area of the quantitation ion for the compound being measured

C(is) = Concentration of the specific internal standard

A(is) = Area of the quantitation ion for the specific internal standard

C(x) = Concentration of the compound being measured

Calculate the relative standard deviation:

$$\%\ RSD = \frac{SD}{\overline{RRF}} \times 100$$

Where:

$$SD = \sqrt{\frac{\sum (RF - avg\ RF)^2}{n-1}} \quad and \quad \overline{RRF} = \frac{\sum RRF}{n}$$

Calculate the 1st Order (linear regression: Y = M(X) + B

Where:

Y = Conc Std

Conc Inst

X = Area Std

Area Istd

M = 1st degree slope

B = Y intercept

Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

C(c) = Measured concentration using selected quantification method

Quantitative Calculations

Waters:

$$Concentration\ (\mu g/L) = \frac{A(x) \times I(is)}{A(is) \times \overline{RRF}\ V(o)}$$

Where:

A(x) = Area of the quantitation ion for the compound to be measured

I(is) = Amount of internal standard added to the water sample (in micrograms)

A(is) = Area of the quantitation ion for the appropriate internal standard

$(\overline{RRF})$ = Average relative response factor for the current initial calibration

V(o) = Original water sample volume (in liters)

Soils:

$$Concentration\,(\mu g/kg) = \frac{A(x) \times I(is)}{A(is) \times \overline{RRF}\,V(g)}$$

Where:

A(x) = Area of the quantitation ion for the compound to be measured

I(is) = Amount of internal standard added to the water sample (in micrograms)

A(is) = Area of the quantitation ion for the appropriate internal standard

$(\overline{RRF})$ = Average relative Response factor from the current initial calibration

V(g) = Original soil sample weight (in grams)

Calculation of the % Error:

$$\%\ Error = \frac{x(i) - x'(i)}{x(i)} \times 100$$

Where:

x(i) = Measured amount of analyte at calibration level *i*, in mass or concentration units
x(i) = True amount of analyte at calibration level *i*, in mass or concentration units

Statistical Information/Method Performance

The LCS/MS and surrogate recoveries and RPD are compared to statistically generated limits for acceptance criteria. The current data is stored in the LIMS under the specific analysis numbers. The historical data for MDLs, MS/MSD, LCS/D, and measurement of uncertainty is reviewed at least annually and updated if necessary. Refer to the QA/QC section of this SOP and the criteria listed throughout this procedure for additional information on the performance of this method.

Quality Assurance/Quality Control

Each extraction batch must contain at least a method blank, laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), and a matrix spike duplicate (MSD) or a laboratory control sample/laboratory control sample duplicate (LCS/LCSD). The spiking solution

contains all analytes of interest. Additional QC samples may be required to meet project or state requirements.

1. The method blank will be evaluated to determine if any contamination exists. Contamination can occur during the extraction process or during analysis. If PAHs are detected in the method blank above the reporting limit (which may be the MDL or the LOQ depending on each client's reporting requirements), any samples with detections of these same PAHs may need to be re−extracted. Analyze a solvent blank using the same internal standard lot to verify that the analytical system and internal standard solution are free from contamination. Reanalyze the batch blank and if found to be free from contamination, re-inject all samples which contain a positive identification of the same compound. If the batch blank confirms the presence of the contaminant report any sample which contains a positive detection at a concentration greater than or equal to ten (10) times the concentration in the blank. Any samples which contain a detection less than ten (10) times the concentration in the blank must be re−extracted and reanalyzed. Phthalates may be detected in the method blank and data reported as long as the phthalate detection is < LOQ. A Nonconformance Form Q-EQA-FRM6869 must be generated and submitted to a supervisor if noncompliant data will be reported.

2. The LCS/LCSD are analyzed to determine the precision of the extraction process. The recoveries of the spiked PAHs should be within the established recovery windows. The established recovery windows are statistically generated and reviewed twice a year. If the recovery for any reported PAH is below QC windows all affected samples will need to be re-extracted. If recoveries are above QC limits, data may be usable if the compounds that recovered above QC limits are not detected in samples. A Nonconformance Form must be generated and submitted to a supervisor.

3. All samples are spiked with 3 surrogate compounds (1-methylnaphthalene-d10, benzo(a)pyrene-d12, and fluoranthene-d10) to evaluate the extraction process. Surrogate windows should be within the established statistical windows. If surrogate recovery does not meet specifications, the samples may need to be re−extracted and reanalyzed.

4. Each sample extract is spiked with an internal standard mix prior to analysis. The internal standard areas from each extract are compared to the internal standard areas from the ICAL. The extract areas must be within −50% to +100% of the ICAL areas. If these criteria are not met, the extract must be reinjected.

5. The lower limit of quantitation (LLOQ) is verified annually through the extraction and analysis of an LCS at 0.5 to 2 times the established LLOQ. The LLOQ is performed on similar analytical instruments such that all are included, at a minimum, within a 3 year time period. Until the laboratory has sufficient data to determine the acceptance limits, the LLOQ criteria is
± 20% of the statistically determined LCS limits.

Figure 1

Mixes

Absolute PAH Standard

acenaphthene
acenaphthylene
anthracene
benzo(a)anthracene
benzo(a)pyrene
benzo(b)fluoranthene
benzo(g,h,i)perylene
benzo(k)fluoranthene
carbazole
chrysene
dibenz(a,h)anthracene
fluoranthene
fluorene
indeno(1,2,3-cd)pyrene
naphthalene
phenanthrene
pyrene
2-Methylnaphthalene
1-Methylnaphthalene
Perylene
Biphenyl
Decalin

Benzo(e)pyrene
pentachlorophenol
Dibenzofuran
Dibenzothiophene
2,6-Dimethylnaphthalene
1-Methylphenanthrene
Thianaphthene
2,3,5-Trimethylnaphthene

Surrogates

1-methylnaphthalene-d10
Benzo(a)pyrene-d12
Fluoranthene-d10

Internal Standards Mix

1,4-dichlorobenzene-d4
naphthalene-d8
acenaphthene-d10
phenanthrene-d10

chrysene-d12
perylene- d12

Table I

DFTPP Key Ion Abundance Criteria (8270C)

Mass	Ion Abundance Criteria
51	30% to 60% of mass 198
68	less than 2% of mass 69
70	less than 2% of mass 69
127	40% to 60% of mass 198
197	less than 1% of mass 198
198	base peak, 100% relative abundance

199	5% to 9% of mass 198
275	10% to 30% of mass 198
365	greater than 1% of mass 198
441	present but less than mass 443
442	greater than 40% of mass 198
443	17% to 23% of mass 442

Table II

DFTPP Key Ion Abundance Criteria (8270D)

Mass	Ion Abundance Criteria
51	10% to 80% of mass 198
68	less than 2% of mass 69
70	less than 2% of mass 69
127	10% to 80% of mass 198
197	less than 2% of mass 198
198	base peak, or >50% mass 442
199	5% to 9% of mass 198
275	10% to 60% of mass 198
365	greater than 1% of mass 198
441	present but less than 24% of mass 442
442	Base peak, or >50% of mass 198
443	15% to 24% of mass 442

Table III

Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification (8270D)

Semivolatile Compounds	Minimum Response Factor (RF)
Naphthalene	0.700
2-Methylnaphthalene	0.400
Dimethylphthalate	0.010
Acenaphthylene	0.900
Acenaphthene	0.900
Dibenzofuran	0.800

Diethylphthalate	0.010
Fluorene	0.900
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Phenanthrene	0.700
Anthracene	0.700
Di-n-butylphthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Benzo(a)anthracene	0.800
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octlyphthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
Benzo(g,h,i)perylene	0.500

Table IV

Semivolatile Internal Standard with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
1,4-Dioxane	Quinoline	Acenaphthene
Benzenethiol	2-Methylnaphthalene	Acenaphthylene
N-Nitrosodimethylamine	Naphthalene	Diethyl phthalate
	Bis(2-chloroethyl)ether	Dimethyl phthalate
	1-Methylnaphthalene	Fluorene
	1-Methylnaphthalene-d10 (surr)	Dibenzofuran

Phenanthrene-d_{10}
Anthracene
Phenanthrene
Di-n-butyl phthalate
Fluoranthene
Hexachlorobenzene
1,2-Diphenylhydrazine
Fluoranthene-d10 (surr)

Chrysene-d_{12}
Pyrene
Benzo(a)anthracene
Bis(2-ethylhexyl) phthalate
Butyl benzyl phthalate
Chrysene

Perylene-d_{12}
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Benzo(a)pyrene-d12 (surr)
Dibenz(a,h)anthracene
Indeno(1,2,3-cd)pyrene
Di-n-octylphthalate
Benzo(e)pyrene
Perylene

(surr) = surrogate

End of document

Version history

Version	Approval	Revision information
12	08.SEP.2016	
13	15.AUG.2017	

eurofins	Always check on-line for validity **Semivolatile Organic Compounds by Method 8270D in Aqueous and Non-Aqueous Matrices using GC-MS**	Level: **Work Instruction**
Document number: **T-SVOA-WI9617**		
Old Reference: **1-P-QM-WI-9015100**		
Version: **8**		Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **22-AUG-2017**	Document users: **5_EUUSLA_GC/MS Semivolatiles_All**	Responsible: **5_EUUSLA_GC/MS Semivolatiles_Manager**

Revision Log

Revision: 8		**Effective Date:** This version
Section	**Justification**	**Changes**
References	Most current version	Added reference for Method 8000D
Procedure F	Enhancement	Added % Error verification for initial calibration levels
Calculations	Enhancement	Added calculation for % Error
Quality Assurance/ Quality Control	Enhancement	Added LLOQ information

Revision 7	**Effective Date:** Apr 13, 2016	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout document	Formatting requirement	Moved all calculations to the Calculations section
Reference	Updated to current version	Changed Method 8000B to Method 8000C and updated revision number and effective date
Basic Principles	Reflects current practices	Changed 1-microliter to 0.5 or 1-microliter to cover the range currently used
Reagents and Standards C.2 and C.3	Enhancement	Added 250 mL aqueous analysis requirement;
Reagents and Standards C.2	Formatting	Changed 0º to 6ºC to 0º to 6ºC ,not frozen
Procedure A	Reflects current practices	Changed Standard prep from every week to10 days to every 20-30 days
Procedure B and E	Enhancement	Added 250 mL aqueous analysis requirements
Procedure F.3 table	Correction	Changed the requirement of the height of the valley between two isomer peaks to 25% of the sum of the two peak heights
Procedure F.3 table footnote	Update to current version	Changed USEPA Method 8000B to USEPA Method 8000C
Procedure G table	Correction	Changed the requirement of the height of the valley between two isomer peaks to 25% of the sum of the two peak heights
Quality Assurance /Quality Control table	Enhancement	Added allowance for high bias in LCS if compounds are non-detects in samples

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8270D, Rev. 4, February 2007.
2. Test Methods for Evaluating Solid Wastes, SW-846 Method 8000C, Rev. 3, March 2003.
3. Test Methods for Evaluating Solid Wastes, SW-846 Method 8000D, Rev. 4, July 2014.
4. *Federal Register*, Vol. 57, No. 227, November 24, 1992, p. 55114 (TCLP).
5. *Federal Register*, Vol. 57, No. 160, August 18, 1992, p. 37203 (CCW).
6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Q-EQA-FRM6869	Nonconformance Form
T-SVOA-WI9598	GC/MS Preventative and Corrective Maintenance
T-SVOA-WI11998	Semivolatile Spiking and Calibration Standards
QA-SOP11178	Demonstrations of Capability

Scope

This method is suitable for the determination of the concentration of certain semivolatile organic compounds (priority pollutant list, target compound list, Appendix IX list, TCLP list, and CCW list) found in soils, tissues, waters, and leachates. Typical limits of quantitation (LOQ) achieved are 33 µg/kg for soils, 132 µg/kg for tissues, 1 µg/L for waters and 0.002 mg/L for leachates. Specific compound lists and associated method detection limits (MDLs) and LOQ can be found in the Laboratory Information Management System (LIMS) under the analysis numbers listed in the header of this SOP.

Basic Principles

A 0.5 or 1−microliter mixture of organic compounds in methylene chloride is injected onto a fused silica capillary column coated with a relatively non−polar stationary phase, which is enclosed in a temperature controlled oven. A carrier gas, ultra pure helium, passes continuously through the column. The GC oven is temperature programmed and the organic mixture separates into its individual components as it moves along the length of the column. This separation is a function of the polarity and boiling point of the individual compounds. The column empties into a mass selective detector. When a compound reaches the detector, it is bombarded by high energy electrons (70 eV). This causes the compounds to fragment, forming ions. By applying various voltages to lenses in the area where the ions are formed, the positive ions are thrust into a quadrupole mass analyzer, which selects for a given mass fragment at a given time. These selected fragments reach an electron multiplier, which detects and generates a signal for each mass fragment. The signals are amplified and sent to a computer making storage and manipulation of the data possible. Target compounds are identified on the basis of relative retention times and spectral match to standards. Standards are injected every 12 hours on each system used for analysis.

Quantification is achieved via use of the internal standard calibration technique. The average relative response factor of a multi−point calibration is used for quantification when the appropriate criteria are met.

Interferences

Method interferences may be caused by impurities in solvents, reagents, and glassware, or other hardware used in the processing of samples. All glassware is solvent rinsed before use and a method

blank is performed with each extraction batch to demonstrate that the extraction system is free of contamination.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound and reagent should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means are available such as fume hoods, safety glasses, lab coats, and gloves.

All solvent waste generated from this analysis must be collected for recycling (if applicable) or must be disposed of in designated containers. These are then transferred to a lab–wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) must be disposed of in the normal solid waste collection containers or sharps containers, as applicable.

Personnel Training and Qualifications

Education Requirement: Degree in science or relevant experience.

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently perform daily maintenance, column and source changing procedures, calibration techniques, interpret chromatograms, calculation, data review, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample. Refer to QA-SOP11178 for more guidance on these options.

Sample Collection, Preservation, and Handling

Water samples may be preserved with sodium thiosulfate ($Na_2S_2O_3$) and must be extracted within 7 days of collection. Solid samples are not preserved and must be extracted within 14 days of collection.

Samples are stored at 0º to 6ºC, not frozen. Extracts must be analyzed within 40 days of extraction and are stored in amber vials at ≤ -10C (freezer).

Apparatus and Equipment

1. 25−µL syringe

2. Hewlett−Packard Model 5890 (Series I and II) or Hewlett-Packard/Agilent 6890 Gas Chromatograph or equivalent

3. Hewlett−Packard Models 5971, 5972, and Hewlett-Packard/Agilent 5973, 5975 Mass Selective Detector or equivalent

4. Thru-Put Systems Target Acquisition Software/Oracle Database or equivalent

Reagents and Standards

A. Standard/spiking concentration and reagent vendors are subject to change without notification.

B. Reagents

1. Methylene chloride, pesticide grade. Store at room temperature.

2. UPC (ultra pure carrier) helium for carrier gas.

C. Standards

1. Documentation of all stocks and calibration standards is maintained in the Standards Database (electronic standard preparation notebook) or in a standards preparation logbook.

2. 50 ng/µL (12.5 ng/µL for 250 mL aqueous analyses) Solution of decafluorotriphenylphosphine (DFTPP) containing pentachlorophenol, benzidine and DDT, prepared from Absolute Standards, Inc., Part #43030 in methylene chloride or equivalent. Store at 0º to 6ºC, not frozen, for up to 6 months.

3. Supelco Equity Semivolatile Internal Standard Mix, Part #46955-U or equivalent, 1000 µg/mL in methylene chloride. Ampulated solutions are maintained under refrigeration (0º to 6ºC, not frozen) until consumed or manufacturer determined expiration date. Working solution is maintained at room temperature and is replenished daily from ampulated solutions. For 250 mL aqueous analyses Supelco Equity Semivolatile Internal Stanadard Mix is diluted 4x to make a 250 µg/mL in methylene chloride solution. This solution is maintained under refrigeration (0º to 6ºC, not frozen) for up to 6 months.

4. Refer to *T-SVOA-WI11998* for the preparation and storage of calibration, check, and spiking solutions.

Calibration

See Procedure F for initial calibration processing and Procedure G for continuing calibration check processing.

Procedure

A. Standard preparation – These solutions are used to standardize the GC/MS system every 12 hours and are prepared approximately every 20-30 days or more frequently if needed based on consumption. See *T-SVOA-WI11998* for standard preparation. Calibration standard solutions may be used up to the labeled expiration date or until component degradation is observed.

B. Internal standard mix is added to all standards and subsequent samples at a concentration of 20 µg/mL (5 µg/mL for 250 mL aqueous analyses). Using a 25-µL syringe, 20 µL of Supelco Equity Semivolatile Internal Standard Mix or equivalent, 1000 µg/mL (250 µg/mL for 250 mL aqueous analyses) in methylene chloride are added to the 1 mL of standard or sample extract.

C. Daily maintenance – Refer to *T-SVOA-WI9598* for this procedure.

D. Instrument conditions

Equip a GC/MS (such as referenced under Apparatus and Equipment) in one of the two following manners:

1. For a 5890/5971 or 5972 and 6890/5973 or 5975

   a. Column – 30M × 0.25 mm ID, 1.0 um df, J&W Scientific DB-5MS or equivalent

   b. Injector – Split/splitless operated in splitless mode

   c. Injector Temp – 275°C

d. Detector Temp – 300°C

e. Gas – Helium at approximately 1.5 mL/min, constant flow mode

f. Oven Temp – 45°C for 3 minutes, ramp at 8°C/minute to 225°C, then ramp at 12°C/minute to 300°C and hold for 7.5 minutes.

2. For a 6890/5973 or 5975

a. Column – 20M × 0.18 mm ID, 0.18 um df, J&W Scientific DB-5MS or equivalent

b. Injector – Split/splitless operated in split mode, 30:1 split

c. Injector Temp – 275°C

d. Detector Temp – 280°C

e. Gas – Helium at approximately 1.0 mL/min, constant flow mode

f. Oven Temp – 40°C for 1 minute, ramp at 25°C/minute to 100°C, then ramp at 30°C/minute to 280°C, followed by another ramp at 25°C/minute to 320°C, hold for 2 minutes.

NOTE: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions that give the performance required by the method are acceptable.

E. Tuning

The GC/MS must be tuned using a 50 ng/µL (12.5 ng/µL for 250 mL aqueous analyses) solution of DFTPP containing pentachlorophenol, benzidine, and DDT.

Frequency	Acceptance Criteria	Corrective Action
Every 12 hours	1. Criteria in Table I 2. DDT breakdown ≤20%* 3. Tailing factors: • Benzidine ≤2 • Pentachlorophenol ≤2	1. Retune. Analysis cannot proceed until tune meets criteria 2. More aggressive injection port maintenance 3. Clean the source 4. Change the column

*__NOTE:__ DDT breakdown greater than 20 percent may be acceptable if you are calibrating for polynuclear aromatic hydrocarbon compounds only. Consult supervisor when this situation occurs.

1. Use only the background−subtracted spectrum of the following when evaluating the DFTPP:

A three scan average of the apex of the scan, the apex of the scan –1 and the apex of the scan +1.

NOTE: All standards, samples, and associated quality control samples with a particular tune must use the identical conditions of the mass spectrometer.

2. Calculation of DDT breakdown

Where:

DDE and DDD = The breakdown products of DDT

TIC = Total Ion Chromatogram

F. Initial calibration (ICAL)

1. Perform standardization by analyzing at least six levels of calibration standards ranging from 0.5 µg/mL to 120 µg/mL. (Refer to *T-SVOA-WI11998* for the preparation of calibration standards.) Use the internal standard calibration technique to generate an average relative response factor for each compound. Table II lists the six internal standards used for the method and the target compounds that are associated with each internal standard.

2. A method detection limit (MDL) standard must be analyzed with each initial calibration. This standard is prepared at the departmental MDL and is not to be included in the calibration curve. All compounds must be detected in the MDL standard.

3. Initial Calibration Verification (ICV) standard is also analyzed with each initial calibration. The ICV is made from a second source standard and has an acceptance window of 70% to 130%.

Frequency	Acceptance Criteria	Corrective Action
Initially and when the daily calibration standard fails criteria. Initially establish with at least six levels of standards and an MDL standard.	1. Minimum response factors must be met in all levels of the calibration – especially in the lowest level of the calibration (see table IV). 2. %RSD for each compound should be less than or equal to 20%. If more than 10% of the compounds in the ICAL exceed 20% RSD and/or also do not meet the minimum correlation coefficient for alternate fits (ie, the correlation coefficient is	1. Any target analyte with an %RSD of ≥20% should use the average RRF. For any analyte in which the %RSD >20%, use a first degree (linear) fit if the correlation coefficient is ≥0.99. If the CC of the linear fit is <0.99, then a second order (quadratic) fit may be used provided the coefficient of determination is ≥0.99. If both the CC for the linear fit and the COD for the quadratic fit are ≥0.99 for any given analyte, then use the fit with the smallest negative y-intercept. When

	<0.990) then the analysis cannot proceed. 3. The % Error is measured between the calculated and expected amounts of an analyte at each calibration level to determine the calibration function acceptability for linear and non-linear curves. The % Error should be ≤ 30% for all standards. For some data uses, ≤ 50% may be acceptable for the lowest calibration level. 4. All compounds of interest must be detected in the MDL standard. 5. The relative retention times of the target compounds must agree within 0.06 relative retention time units. The exception would be in the case of system maintenance. 6. Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.	using a quadratic fit, if the y-intercept quantifies to be greater than the MDL, consult your supervisor immediately or recalibrate. See below for corrective action if the coefficient of determination (COD) for a quadratic fit is <0.99.** 2. The % Error is measured between the calculated and expected amounts of an analyte at each calibration level to determine the calibration function acceptability for linear and non-linear curves. The % Error should be ≤ 30% for all standards. For some data uses, ≤ 50% may be acceptable for the lowest calibration level. . 3. If a compound is not detected in the MDL standard, then report to the level of the lowest standard detected. All compounds manually integrated in this standard must be checked for in each sample analyzed under this initial calibration.* 4. More aggressive system maintenance, and recalibrate

*If these situations occur, your supervisor is to be consulted immediately.

**See USEPA Method 8000C for the calculations associated with non-linear fit types.

With supervisory approval, the following problematic compounds can be allowed to fail the 0.99 coefficient of determination criteria for a quadratic fit:

1,4-Phenylenediamine
4-Aminobiphenyl
3,3'-Dimethylbenzidine
4,4'-Methylenebis(2-chloroaniline)
4-Nitroquinoline-1-oxide
1,4-Naphthoquinone
methapyrilene

If the COD is less than 0.99 for any other compound, the system should be inspected for problems and recalibrated. Supervisory approval is required for exceptions to these guidelines.

NOTE: Quadratic fits are not permitted when analyzing samples from South Carolina.

G. Continuing Calibration Verification (CCV)

Frequency	Acceptance Criteria	Corrective Action
1. Every 12 hours 2. Check standard area analyzed at the 30 µg/mL concentration or the fifth level of the calibration.	1. Target analytes should meet the minimum response factor criteria (see Table IV). 2. The maximum % drift for all target analytes is 20%. No more than 20% of the target analytes can be greater than 20% drift. All target analytes that exceed 20% drift must be less than or equal to 50% drift. 3. The relative retention times of the target compounds must agree within 0.06 relative retention time units. The exception would be for the case of system maintenance. 4. The EICP area for each internal standard must fall within the window of -50% to +100% from the areas of the mid-level standard produced during the last initial calibration. 5. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the ICAL as well as the check standard.	1. More aggressive system maintenance 2. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values. 3. Recalibrate

In the event that two consecutive continuing calibration check standards fail for the list of target analytes being quantified, then after the appropriate system maintenance has been performed, two consecutive continuing calibration check standards must pass criteria, before analysis can continue. If the analytical system cannot pass two consecutive checks, then the system must be recalibrated.

H. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The sample component relative retention time must compare within ±0.06 RRT units of the RRT of the standard component.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The primary and secondary ions can be found in Table III.

NOTE: N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. For this reason, it is acceptable to report the combined result for both n-nitrosodiphenylamine and diphenylamine for either of these compounds as a combined concentration.

NOTE: 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene. Given the stability problems, it would be acceptable to calibrate for 1,2-diphenylhydrazine using azobenzene. Under these poor compound separation circumstances the results for either of these compounds should be reported as a combined concentration.

I. Quantitative analysis

Once a compound has been identified, quantitation is based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. The list of primary characteristic ions is listed in Table III. See Calculations section for concentration calculations.

Calculations

A. Calibration calculations

1. Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Area of the characteristic ion for the compound being measured

A(is) = Area of the characteristic ion for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression equation

1st Order (linear) regression: $Y = M(X) + B$

2nd Order (quadratic) regression: $Y = B + M(X) + CX2$

Where:

$Y = \frac{\text{Conc Std}}{\text{ConcInst}}$

$X = \frac{\text{Area Std}}{\text{AreaIstd}}$

M = 1st degree slope

C = 2nd degree slope

B = Y intercept

3. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

C(c) = Measured concentration using selected quantification method

4. Calculation of the percent relative standard deviation (%RSD):

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

Where:

SD = Standard deviation

$\overline{RF}$ = Average response factor

B. Concentration calculations

1. Waters:

$$Concentration\ (\mu g/L) = \frac{A(x) \times I(s) \times V(t) \times D_f}{A(is) \times RRF \times V(o) \times V(i)}$$

Where:

A(x) = Area of characteristic ion for compound being measured

I(s) = Amount of internal standard injected (ng)

V(t) = Volume of concentrated extract in microliters (µL)

Df = Dilution factor

A(is) = Area of characteristic ion for the internal standard

RRF = Relative response factor for the compound being measured

V(i) = Volume of extract injected (µL)

V(o) = Volume of water extracted (mL)

2. Soils:

$$Concentration\ (\mu g/kg) = \frac{A(x) \times I(s) \times V(t) \times G \times D_f}{A(is) \times RRF \times W(s) \times V(i) \times D}$$

Where:

A(x) = Area of characteristic ion for compound being measured

I(s) = Amount of internal standard injected (ng)

V(t) = Volume of concentrated extract in microliters

Df = Dilution factor

A(is) = Area of characteristic ion for the internal standard

RRF = Relative Response factor for the compound being measured

V(i) = Volume of extract injected (µL)

W(s) = Weight of sample extracted or diluted in grams

D = The percent solids (100 - % moisture)/100

G = 1 if extract did not require GPC cleanup

G = 2 if extract required GPC cleanup

C. QC Calculations

1. Percent Recovery:

% Recovery = Concentration found ÷ Concentration spiked x 100

2. Calculations for MS/MSD:

Matrix spike recovery = SSR x SR ÷ SA x 100

Where:

SSR = Spike sample result

SR = Sample result

SA = Spike added

3. Relative Percent Difference (RPD):

$$RPD = \{|MSR \times MSDR| \div \tfrac{1}{2}(MSR \times MSDR)\} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Dup Recovery

Calculation of the % Error:

$$\%\ Error = \frac{x(i) - x'(i)}{x(i)} \times 100$$

Where:

x(i) = Measured amount of analyte at calibration level *i*, in mass or concentration units

x(i) = True amount of analyte at calibration level *i*, in mass or concentration units

Statistical Information/Method Performance

The LCS/MS and surrogate recoveries and RPD are compared to statistically generated limits for acceptance criteria. The current data is stored in the LIMS under the analysis numbers listed in the header of this SOP. The historical data for MDLs, MS/MSD, LCS/D, and measurement of uncertainty is reviewed at least annually and updated if necessary. Refer to the Quality Assurance/Quality Control section of this SOP and the criteria listed throughout this procedure for additional information on the performance of this method.

Quality Assurance/Quality Control

Each extraction batch must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD) or a laboratory control sample/laboratory control sample duplicate (LCS/LCSD). Refer to *T-SVOA-WI11998* for the preparation of quality control spikes. Additional QC samples may be required to meet project or state certification requirements.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards	1. Peak area within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If reinjection meets the criteria, report this injection. 3. If reinjection still shows same problem, report first injection and qualify data with a comment.
Method Blank	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All target compounds must be less than the reporting limit for the associated samples.	1. Inspect system for possible problems and reanalyze. 2. If the surrogates are out of spec high data can be used. (Unless project requirements dictate otherwise).* 3. If the method blank contains target analytes and the associated samples do not contain these compounds, no corrective action is required. If the target compounds in the blank are also in the associated samples, the samples should be reextracted unless it does not interfere with project data requirements.
Laboratory Control Sample/Laboratory Control Sample Duplicate	All percent recoveries within QC limits. Refer to the GC/MS Semivolatile SOP manual for QC windows. These are reviewed and updated on a semiannual basis.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD still out of spec, consult supervisor immediately. Samples may need to be re-extracted. 4. If recoveries are above QC limits, data may be usable if the compounds that recovered above QC limits are not detected in samples.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1. % Recoveries within QC limits. Refer to the GC/MS Semivolatile SOP manual for QC windows. These are reviewed and updated on a semiannual basis. . 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries or RPDs out of spec, consult supervisor.

Quality Control Item	Acceptance Criteria	Corrective Action
Surrogates	All recoveries must be within QC limits. Refer to the GC/MS Semivolatile SOP manual for surrogate windows. These are updated on a semiannual basis.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. If no problem is found, reextract and reanalyze. If surrogates are out of spec high and no targets are detected in the sample no corrective action is required.

*Requires approval of supervisor and completion of Non-Conformance *Q-EQA-FRM6869*.

The lower limit of quantitation (LLOQ) is verified annually through the extraction and analysis of an LCS at 0.5 to 2 times the established LLOQ. The LLOQ is performed on similar analytical instruments such that all are included, at a minimum, within a 3 year time period. Until the laboratory has sufficient data to determine the acceptance limits, the LLOQ criteria is ± 20% of the statistically determined LCS limits.

A. Dilution Criteria

1. Initial dilutions:

a. More than three internal standard areas are less than −50%.

b. Either of the last two internal standard areas are less than −80%.

c. Prescreen data or analyst's judgement of a sample extract's color or viscosity, indicate a possible matrix interference.

2. Secondary dilutions:

Are required to bring all target compounds in the calibration range of the GC/MS.

Table I

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria

51	10% to 80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	10% to 80% of mass 198
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5% to 9% of mass 198
275	10% to 60% of mass 198
365	>1% of mass 198
441	Present but less than 24% of mass 442
442	Base peak, or >50% of mass 198
443	15% to 24% of mass 442

Table II
Semivolatile Internal Standard with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Acrylamide		
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethylphenylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d5 (surr)	1-Naphthylamine
N-Nitroso-di-n-propyl amine	2-Nitrophenol	2-Naphthylamine

Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d6 (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
1,4-Dioxane	1-Methylnaphthalene	4-Nitrophenol
Pyridine	O,O,O-triethylphosphorothioate	Pentachlorobenzene
		1,2,3,4-Tetrachlorobenzene
		1,2,3,5-Tetrachlorobenzene
Acetophenone	Hexachlorpropene	1,2,4,5-Tetrachlorobenzene
o-Toluidine	1,4-Phenylenediamine	2,3,4,6-Tetrachlorophenol
N-Nitrosomethylethylamine	Safrole	2,4,6-Tribromophenol (surr)
N-Nitrosodiethylamine	(2-Bromoethyl)benzene	2,4,6-Trichlorophenol
N-Nitrosopyrrolidine	Caprolactam	2,4,5-Trichlorophenol
N-Nitrosomorpholine	1, 3, 5 – Trichlorobenzene	1,1'-Biphenyl
N,N-dimethyl formamide	1, 2, 3 – Trichlorobenzene	Diphenyl ether
N,N-dimethyl acetamide	1, 2, 3, 4 – Tetrachlorobenzene	Isosafrole
Benzaldehyde	1 – Chloro-4-Nitrobenzene	1,4-Naphthoquinone
	2-chlorobenzaldehyde	1,4-Dinitrobenzene
		1,3-Dinitrobenzene
		Thionazin
		5-Nitro-o-toluidine
(surr) = surrogate		2-chlorobenzalmalononitrile

Table II (continued)

Phenanthrene-d_{10}	Pyrene-d_{10}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Fluoranthene	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Hexachlorobenzene	p-Dimethylaminoazobenzene	Indeno(1,2,3-cd)pyrene
N-Nitrosodiphenylamine	Pyrene	Di-n-octylphthalate
Octachlorostyrene		
Pentachlorophenol	Terphenyl-d_{14} (surr)	3-Methylcholanthrene
Pentachloronitrobenzene	7,12-Dimethylbenz(a)anthracene	
Phenacetin	Chlorobenzilate	
Phenanthrene	2-Acetylaminofluorene	
Pronamide	3,3'-Dimethylbenzidine	
1-Nitronaphthalene	4,4'-Methylenebis(2-Chloroaniline)	
1,2-Diphenylhydrazine	3-Quinuclidinyl benzilate	
Carbazole		
Tetraethyldithiopyrophosphate		
1,3,5-Trinitrobenzene		
Diallate trans/cis		

Phorate
Dimethoate
Methyl parathion
Parathion
4-Nitroquinoline-1-oxide
Methapyrilene
Isodrin
Atrazine

(surr) = surrogate
Benzophenone

Table III
Characteristic Ions for Semivolatile Compounds

1.1.1 Compound	Primary Ion	Secondary Ions
2-Picoline	93	66,92
Aniline	93	66,65
Phenol	94	65,66
Bis(2-chloroethyl) ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene-d_4 (IS)	152	150,115
1,4-Dichlorobenzene	146	148, 113
Benzyl alcohol	108	79,77
1,2-Dichlorobenzene	146	148, 113
N-Nitrosomethylethylamine	88	42,43,56
Bis(2-chloroisopropyl) ether	45	77, 121, 79
Methyl methanesulfonate	80	79,65,95
N-Nitrosodi-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
N-Nitrosodiethylamine	102	42,57,44,56
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	107	122, 121
Bis(2-chloroethoxy)methane	93	95,123
Benzoic acid	105	122,77
2,4-Dichlorophenol	162	164,98

Ethyl methanesulfonate	109	79,97,45,65
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d_8 (IS)	136	68
Naphthalene	128	129,127
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141, 115
2-Methylphenol	108	107,77,79,90
Hexachloropropene	213	211, 215, 117, 141
Hexachlorocyclopentadiene	237	235,272
N-Nitrosopyrrolidine	100	41,42,68,69
Acetophenone	105	71,51,120
4-Methylphenol	108	107,77,79,90
2,4,6-Trichlorophenol	196	198,200

Table III (continued)

1.1.2 Compound	Primary Ion	Secondary Ions
o-Toluidine	106	107,77,51,79
3-Methylphenol (as 4-Methylphenol)	108	107,77,79,90
2-Chloronaphthalene	162	127,164
N-Nitrosopiperidine	114	42,55,56,41
1,4-Phenylenediamine	108	80,53,54,52
1-Chloronaphthalene	162	127,164
2-Nitroaniline	138	92, 65
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
2,6-Dinitrotoluene	165	63,89, 121
Phthalic anhydride	104	76,148
3-Nitroaniline	138	108,92
Acenaphthene-d_{10} (IS)	164	162,160
Acenaphthene	153	154, 152
2,4-Dinitrophenol	184	63, 154, 107
2,6-Dinitrophenol	162	164,126,98,63
4-Chloroaniline	127	129,65,92
Isosafrole	162	131,104,77,51
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89, 182
4-Nitrophenol	109	139,65
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	104,102,76,130
Diethyl phthalate	149	177,150

Fluorene	166	165,167
N-Nitrosodi-n-butylamine	84	57,41,116,158
4-Chlorophenyl phenyl ether	204	206,141
4,6-Dinitro-2-methylphenol	198	51, 105, 182, 77
N-Nitrosodiphenylamine	169	168,167
Safrole	162	104,77,103,135
Diphenylamine	169	168,167
1,2,4,5-Tetrachlorobenzene	216	214,179,143,218
1-Naphthylamine	143	115,89,63
4-Bromophenyl phenyl ether	248	250,141
2,4,5-Trichlorophenol	196	198,97,132,200
Hexachlorobenzene	283	142,249
Pentachlorophenol	266	264,268
5-Nitro-o-toluidine	152	77,79,106,94
Thionazin	107	96,97,143,79

Table III (continued)

1.1.3 Compound	Primary Ion	Secondary Ions
4-Nitroaniline	138	65,108,92,80
Phenanthrene-d_{10} (IS)	188	94,80
Phenanthrene	178	179,176
Anthracene	178	176,179
1,4-Dinitrobenzene	168	75,50,76,92
1,3-Dinitrobenzene	168	76,50,75,92
Diallate (cis or trans)	86	234,43,70
Pentachlorobenzene	250	252,248,215,254
5-Nitro-o-anisidine	168	79,52,138,153,77
Pentachloronitrobenzene	237	142,214,249,295
4-Nitroquinoline-1-oxide	190	160, 116, 114
Di-n-butyl phthalate	149	150,104
2,3,4,6-Tetrachlorophenol	232	131,230,166,234
Fluoranthene	202	101, 203, 100
1,3,5-Trinitrobenzene	213	74,75,120,91
Benzidine	184	92,185
Pyrene	202	101,203
Phorate	75	121,97,93,260
Phenacetin	108	179,109,137,80
Dimethoate	87	93,125,143,229
4-Aminobiphenyl	169	168,170,115
Pronamide	173	175,145,109,147
Dinoseb	211	163,147,117,240

Disulfoton	88	97,89,142,186
Butyl benzyl phthalate	149	91,206
Methyl parathion	109	125,263,79,93
Dimethylaminoazobenzene	225	120,77,148,42
Benz(a)anthracene	228	229,226
Chrysene-d_{12} (IS)	240	120,236
3,3'-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
Parathion	109	97,291, 186
Bis(2-ethylhexyl) phthalate	149	167,279
3,3'-Dimethylbenzidine	212	106,196,180
Methapyrilene	97	58, 72, 191, 261
Isodrin	193	66, 195, 263, 265,
Di-n-octyl phthalate	149	167,43, 150
2-Aminoanthraquinone	223	167, 195, 139
Aramite	185	191,319,334,197,321

Table III (continued)

1.1.4 Compound	Primary Ion	Secondary Ions
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Chlorobenzilate	139	251, 253, 111, 141
Benzo(a)pyrene	252	253,125
Perylene-d_{12} (IS)	264	260,265
7,12-Dimethylbenz(a)anthracene	256	241,239,120
2-Acetylaminofluorene	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	231	266, 140, 195
3-Methylcholanthrene	268	252,253,126,134
Indeno(1,2,3-cd)pyrene	276	138,227
Dibenz(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
1,2-Diphenylhydrazine	77	105, 182, 51
Endosulfan I	195	33
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64, 92
Nitrobenzene-d_5 (surr)	82	128,54
N-Nitrosodimethylamine	74	42,44
Phenol-d_6 (surr)	99	42,71
Terphenyl-d_{14} (surr)	244	122,212
2,4,6-Tribromophenol (surr)	330	332,141

N,N-dimethyl formamide	73	44,42
N,N-dimethyl acetamide	87	72,44,42
(2-Bromoethyl)benzene	184	77,91,105,186
Atrazine	200	173,215
Benzaldehyde	77	105, 106
Caprolacatam	113	55,56
1,1-Biphenyl	154	153,152,76
Carbazole	167	166,139
1,3,5-Trichlorobenzene	180	182,145,109
1,2,3-Trichlorobenzene	180	182,145,109
1,2,3,4-Tetrachlorobenzene	216	214,218,179
1-Chloro-4-Nitrobenzene	157	111,75,99

IS = internal standard
surr = surrogate

Table III (continued)

1.1.5 Compound	Primary Ion	Secondary Ions
Acrylamide	71	55, 44
Octachlorostyrene	308	343, 380, 273
1,2,3,5-tetrachlorobenzene	216	214, 218, 179, 143
1,2,3,4-tetrachlorobenzene	216	214, 218, 179, 143
2-chlorobenzaldehyde	139	111, 140, 76
Benzophenone	105	182, 77, 51
3-Quinuclidinyl benzilate	183	126, 337, 110
2-chlorobenzalmalononitrile	153	188, 126

Table IV
Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Table III

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800

Semivolatile Compounds	Minimum Response Factor (RF)
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050

Table IV (continued)

Semivolatile Compounds	Minimum Response Factor (RF)
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010

2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethylphthalate	0.010
2,6-Drintrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dintirophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4Dinitrotoluene	0.200
Diethylphthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenylether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenylether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100

Table IV (continued)

Semivolatile Compounds	Minimum Response Factor (RF)
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700

Carbazole	0.010
Di-n-butylphthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
3,3'-Bichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octlyphthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Q-EQA-FRM6869 Nonconformance Form
QA-SOP11178 Demonstrations of Capability
T-SVOA-WI11998 Semivolatile Spiking and Calibration Standards
T-SVOA-WI9598 GC/MS Preventative and Corrective Maintenance

End of document

Version history

Version	Approval	Revision information
6	23.MAR.2016	
7	13.APR.2017	
8	15.AUG.2017	

eurofins	Always check on-line for validity **Separatory Funnel Extraction by Method 3510C for BNAs in Wastewater**	Level: **Work Instruction**
Document number: **T-OE-SVOA-WI11432**		
Old Reference: **1-P-QM-WI-9015076**		
Version: **14**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **30-SEP-2016**	Document users: **5_EUUSLA_GC/MS Semivolatiles_Manager, 5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Semivolatile Waters, 6_EUUSLA_GC/MS Semivolatiles_Analysts**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis DOD - 0813, 11010, 10464, 10467, 10476

Revision Log

Revision: 14		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects current analysis numbers Old reference numbers not needed	Added analysis numbers 14239, 14240, 14243, 14245, 14246, 14247, 14241,14242, 14244 Removed old reference numbers
Apparatus and Equipment	Reflects current practice	Added Automated Water Extraction Bench, Rapid-Vap Evaporator, Rapid-Vap Concentrator Tube

Revision: 14	**Effective Date:** This version	
Procedure F. and G.	Reflects current practice	Added surrogate details for new analysis numbers
Procedure M.-U.	Reflects current practice	Separated 1−L extraction from 250−mL extraction and added new procedure for 250−mL extraction

Revision: 13	**Effective Date:** May 9, 2016	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1−P−QM-QMA-9017356	Removed revision logs up to the previous version
Historical/Local Document Number	Reflects current analyses	Removed prep #11015
Throughout Document	Reflects current analyses	Removed analysis #11013 and added analyses #13615 and 13618
Reference	Reflects method referenced in LIMS	Added revision number to SW-846 method
Definitions	Common terms that are defined in higher level document	Removed section and defined first use of each acronym in document
Personnel Training and Qualifications	Reflects current process	Revised language regarding IDOCs and DOCs
Sample Collection, Preservation, and Handling	Reflects current process	Added the temperature for storage of extracts
Reagents and Standards	Reflects current process Reflects current reagents in use	Added the word approximately relative to the temperature of baking sodium sulfate Added reagent water
Procedure N	Clarification	Added step for visual confirmation of adequate phase separation during shake
Procedure O	Clarification	Added that no metal beakers are needed for 250 mL prep
Procedure S	Reflects current practice	Added use of hand held bulb to squeeze excess methylene chloride from sodium sulfate column
Procedure T	Enhancement	Added language regarding rate of concentration

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3510C, Rev 3, December 1996.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0949, 1309, 1476, 1536, 1946, 1947, 1953, 2035, 2395, 4615, 4678, 4688, 6387, 6397, 7804, 7805, 10032, 10723, 10724, 10727, 10728, 13615, 13618	Semivolatile Organic Compounds Including DRO/ORO by Method 8270C in Aqueous and Non-Aqueous Matrices Using GC-MS
Analysis #10461, 10462, 10726	Semivolatile Organic Compounds by Method 8270D in Aqueous and Non-Aqueous Matrices using GC-MS
1−P−QM−PRO−9015475	Glassware Cleaning for Organic Extractions

Document	Document Title
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Purpose
This procedure is used for the extraction of semivolatile compounds by SW-846 Method 3510C in water and wastewater.

Scope
This method is for the extraction of semivolatile organic compounds at low ppb to low ppm levels in water and wastewater matrices that are not prone to emulsions. High levels of organic compounds and/or extreme alkalinity or acidity in the sample often interfere with normal detection limits.

Basic Principles
A sample is placed into a separatory funnel. Surrogate standards are added to each sample to monitor recovery. The pH of the sample is adjusted to >11 and the sample is serially extracted with methylene chloride. The pH is then adjusted to <2 and the sample is again extracted with methylene chloride. The solvent fractions are combined, and the extract is dried and concentrated to 1.0 mL.

Procedural changes to use a reduced sample aliquot while maintaining the default reporting limits are permitted as long as the reagent aliquots are reduced proportionally and a quad study is performed and on file.

Reference Modifications
Acid compounds are added at a concentration of 100 ppm in the matrix spiking and LCS solutions so that their concentrations in the extract are within calibration range.

Surrogate and matrix spiking solutions are not added before the transfer to the separatory funnel for several reasons:

1. Samples must be poured from the amber bottles to determine the matrix and volume of sample to use for each extraction.

2. Many sample bottles have no headspace and there is no room to add surrogate to the sample in the bottle.

3. Due to the volume of samples extracted, a separate graduated cylinder for each sample is unrealistic.

4. To maintain consistency with all extractions, no samples are spiked in the bottle or graduated cylinders.

Interferences

High levels of organic compounds in the sample lead to interferences with normal detection limits.

Impurities in solvents, reagents, glassware, or other hardware used in sample processing lead to interferences with the method. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of sample to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab−wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the employees training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the extraction and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, or one blind sample.

Sample Collection, Preservation, and Handling

Samples must be collected in amber glass bottles with PTFE-lined lids and stored under refrigeration at 0º to 6ºC, not frozen prior to extraction. Samples must be extracted within 7 days of collection. The extract is stored in an amber autosampler vial in the freezer ≤-10 °C for up to 40 days prior to analysis.

Apparatus and Equipment

1. Separatory funnel for extracting organic components from an aqueous matrix

2. Kuderna-Danish (K-D) assembly with appropriate ampule for extracting the solvent used during the extraction

3. Steam bath – VWR/LLI Model #1127 or equivalent

4. Graduated cylinders – Class A, assorted sizes

5. Pipettes – Class A, assorted sizes

6. Pipettes – Disposable

7. Solvent pumps – Beckman, adjustable

8. Balance – Capable of weighing to 0.01 g

9. Automatic shaker – Capable of holding separatory funnels

10. Centrifuge – Beckman GS-6 or equivalent

11. Sodium sulfate columns with extra course frits

12. Micro-snyder columns

13. Wash bottles – Teflon™

14. Amber autosampler vials

15. Teflon™ boiling chips

16. Automated Water Extraction Bench – capable of holding separatory funnels for 250−mL extractions.

17. Rapid-Vap Evaporators – for concentration of 250−mL extractions

18. Rapid-Vap Evaporator Tubes – Class A

Reagents and Standards

1. Methylene chloride – Pesticide grade or equivalent. Store at room temperature for up to one year.

2. 10N Sodium hydroxide (NaOH) – Lab chem or equivalent. Store at room temperature for up to one year.

3. Sulfuric acid – ACS grade or equivalent, concentrated. Store at room temperature for up to one year.

4. Sodium sulfate – Reagent grade or equivalent. Bake at approximately 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. Store in a glass jar for up to 1 year after baking.

5. Reagent water – water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the reagent water supplied at the taps in the laboratory meets this criterion. If the reagent water does not meet the requirements, see your supervisor for further instructions.

6. All QC standards added during extraction process are prepared by Department 4036 using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical Analysis #0949, 1309, 1476, 1536, 1946, 1947, 1953, 2035, 2395, 4615, 4678, 4688, 6387, 6397, 7804, 7805, 10032, 10723, 10724, 10727, 10728, 13615, 13618 14239, 14240,14245, 14246, and 14247 and Analysis #10461, 10462, 10726,14241, and 14242.

See 1−P−QM−PRO−9015490 for storage and handling of spikes.

Preparation of Glassware

See 1−P−QM−PRO−9015475.

Calibration

Not applicable to this procedure

Procedure

A. Determine the volume of sample to be used for each extraction. Ideally, one full bottle is used (Typically this is 1L for routine extraction or 250 mL for mini−extractions).

1. 1 Liter analysis numbers: 0949, 1309, 1476, 1946, 1953, 2395, 4678, 6387, 7805, 10461,13615.

2. 250 mL analysis numbers: 14239, 14240, 14241, 14242, 14246, 14247.

B. If the sample is in a 1-L bottle (or 250−mL bottle) and not a quality control sample [Background (BG), matrix spike (MS), matrix spike duplicate (MSD)], mark the water level on the outside of the sample bottle in order to later determine the sample volume.

1. Shake the bottle vigorously.

2. Pour the sample into a separatory funnel.

3. Record any comments about the sample in the extraction log.

C. If the sample bottle is larger than 1−L (or more than 250−mL for mini-extractions) or the sample is a quality control sample, exactly 1 L (or 250 mL) of sample is extracted.

1. Shake the sample bottle vigorously.

2. Use a clean graduated cylinder to measure the necessary volume of sample and pour it into a separatory funnel.

3. Record the sample volume and any comments about the sample in the extraction log.

4. Use a wash bottle to rinse the graduated cylinder with methylene chloride and add the rinseate to the separatory funnel.

NOTE: Reduced volumes of sample are only used with prior approval by the client or if the matrix of the sample does not allow the extraction to proceed using full volume. The sample must then be diluted to 1 L or 250 mLdepending on the extraction.

D. The method blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) (if applicable) are prepared using 1 L (or 250 mL for mini-extractions) of non−deionized reagent water (tap water) measured into the separatory funnel.

E. The background, MS, and MSD are prepared on three separate aliquots of a field sample.

F. Surrogates

1. For analyses 1309, 1423, 1476, 4678, 6387, 10464 and 13615 - Add 1.0 mL BNA surrogate to all samples, blanks, LCS, LCSD (if applicable), MS, and MSD.

2. For analysis 14239, 14240, 14241, 14242, 14246, 14247 – Add 1.0 mL of Mini BNA surrogated to all samples, blanks, LCS, LCSD(if applicable), MS, and MSD.

NOTE: The standard must drip directly into the aqueous sample without touching the glass side of the separatory funnel to avoid poor recoveries.

G. Spiking solutions

1. For analyses 1423 and 4678 - Add 1.0 mL of LCS spiking solution to the LCS, LCSD (if applicable), MS, and MSD.

2. For analysis1476 – Add 1.0 mL of LCS spiking solution to the LCS, LCSD (if applicable), MS, and MSD. If required, spike 1.0 mL of Benzenethiol spike to a separate LCS and LCSD.

3. For analysis 13615- Add 1.0 mL TPH-DRO spike (or 0.25 mL TPH-DRO spike for 250 mL extraction).

4. For analyses 1309 and 10464 – Add 1.0 mL of LCS & APPIX Mix #1 to the LCS, LCSD (if applicable), MS, and MSD. If required, add 1.0 mL of APPIX Mix#2 to a separate LCS/LCSD.

5. For analysis 6387 – Add 1.0 mL of LCS & APPIX Mix #1 to the LCS, LCSD (if applicable), MS, and MSD.

6. For analysis 14239, 14240, 14242 - Add 1.0 mL of Mini LCS spiking solution to the LCS, LCSD (if applicable), MS, and MSD.

7. For analysis 14247 - Add 1.0 mL of Mini LCS spiking solution to the LCS, LCSD (if applicable), MS, and MSD. If required, spike 1.0 mL of Benzenethiol spike to a separate LCS and LCSD.

8. For analysis 14241, 14246 - Add 1.0 mL of Mini LCS & Mini APPIX Mix #1 to the LCS, LCSD (if applicable), MS, and MSD. If required, add 0.25 mL of APPIX Mix#2 to a separate LCS/LCSD.

NOTE: The standard must drip directly into the aqueous sample without touching the glass side of the separatory funnel to avoid poor recoveries.

H. If the sample requires compounds in addition to the priority pollutant semivolatile compounds, add 1.0 mL (or 0.25 mL for 250−mL extractions) of the 100 ppm spike.

I. Add 10N sodium hydroxide to each sample, Method Blank, LCS/LCSD to adjust the pH to >11.

J. Determine the volume of sample used for extraction:

1. If the original sample bottle is empty:

a. Use a solvent pump to measure 60 mL of methylene chloride for 1 L extractions (15 mL for 250−mL extraction).

b. Rinse the sample bottle by adding the methylene chloride to the bottle. Then cap the bottle and invert several times.

c. Add the solvent to the separatory funnel.

d. Fill the bottle to the marked level with non−deionized reagent water (tap water) and transfer the water to a graduated cylinder to determine the initial volume.

2. Alternatively, tare the empty sample bottle on the balance.

a. Fill the bottle with non−deionized reagent water (tap water) to the marked line and place the bottle back onto the balance.

b. Round the weight to a whole number. This is the volume of sample used for extraction.

3. If the sample container is not empty, use a solvent pump to measure 60 mL of methylene chloride (15 mL for 250−mL extractions) and add the solvent directly to the separatory funnel.

K. Cap the funnel, invert it, and vent immediately.

L. Handshake and vent frequently until the pressure is stable.

M. For analysis numbers 0949, 1309, 1476, 1946, 1953, 2395, 4678, 6387, 7805, 10461,13615 - Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

For analysis numbers 14239, 14240, 14241, 14242, 14246, 14247 – Separatory funnels remain on the Automated Water Extraction bench. Press the green “start” button to lower the hood and activate the Automated Water Extraction Bench. The bench will tumble for 2 minutes. The sash will rise after the samples have tumbled.

NOTE: Shaker speeds vary greatly between instruments; the proper setting is marked on each.

N. For analyses 0949, 1309, 1476, 1946, 1953, 2395, 4678, 6387, 7805, 10461, 13615, Place the separatory funnel on the rack and allow it to sit undisturbed for approximately 10 minutes. For analyses

14239, 14240, 14241, 14242, 14246, 14247, the separatory funnel will sit undisturbed on the Automated Water Extraction Bench. The time required for extracts to set undisturbed is based upon visual confirmation that the layers are adequately separated. Additional time may be necessary for samples with unusual high density (i.e. high salt content).

If an emulsion forms and is >1/3 of the volume of the solvent layer, mechanical techniques such as stirring and centrifugation must be employed to complete the separation.

O. For analyses 0949, 1309, 1476, 1946, 1953, 2395, 4678, 6387, 7805, 10461, 13615, drain the solvent layer into a metal beaker and pour it through approximately 10 cm of sodium sulfate into a K−D apparatus containing a Teflon™−boiling chips. For analyses 14239, 14240, 14241, 14242, 14246, 14247, drain the solvent layer directly into the sodium sulfate column containing approximately 10 cm of sodium sulfate. This drains into a Rapid-Vap evaporator tube.

P. Use a solvent pump to add 60 mL of methylene chloride (15 mL for 250−mL extractions) to the separatory funnel and repeat steps Procedure J. through O., venting only as necessary. The acid extract is also added to this K−D (or evaporator tube for 250−mL extractions).

Q. Add sulfuric acid with a disposable pipette to adjust the sample pH to <2. Using three 60−mL aliquots of methylene chloride (15 mL for 250−mL extractions), serially extract the sample as described in Procedure J. through O.

R. Collect the solvent in a K−D setup (or evaporator tube for the 250−mL extractions). For 1L extractions, rinse the metal beaker with approximately 20 mL of methylene chloride and pour into the sodium sulfate column.

S. Use a wash bottle to rinse the sodium sulfate column with approximately 20 mL of methylene chloride (5 mL for 250−mL extractions). Use a hand held bulb to squeeze any excess methylene chloride from the sodium sulfate column.

T. For 1−L extractions:

1. Attach a 3-ball Snyder column to the K−D, wet with solvent, and concentrate the extract to approximately 1−mL on a steam bath at 80° to 90°C. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. Adjust the vertical position of the apparatus and the water temperature as needed to complete the concentration in 10 - 20 min. Allow the sample to cool for 10 minutes.

NOTE: This steam bath temperature ensures concentration in a reasonable length of time.

2. Attach a microsnyder to the ampule and concentrate on a steam bath to slightly below 1 mL.

3. Bring the combined extract to 1.0 mL using methylene chloride.

4. Determine the final volume by placing the extract into an amber-autosampler vial and comparing the level in the vial to a reference vial containing the exact targeted final volume. Add Methylene chloride to the extract using a disposable pipette until exactly the same level is in both vials.

5. If too much solvent is added to the sample vial, remove the extract from the vial and concentrate it by microsnydering to slightly less than the targeted final volume and rebottle. Cap the extract securely and store in the freezer. Record the final volume in the extraction log.

U. For 250−mL extractions:

1. Place the evaporator tube containing the solvent extract into the pre-heated (approximately 80°C) Rapid-Vap Evaporator. Set the timer for 15 minutes and speed at 75% (the speed is the rotation within the unit while the sample is evaporating). Confirm that the nitrogen is set at 15 PSI (this is adjusted on the regulator attached to the nitrogen line). Allow samples to concentrate to around 0.5 mL. Bring to final volume 1 mL in the Class A Concentrator Tube by rinsing with Methylene Chloride.

NOTE: The temperature, timer, and speed settings can be adjusted as necessary to accommodate difficult sample matrices, etc.

2. Mix the sample thoroughly in the Concentrator Tube and transfer extract to an amber autosampler vial. Cap the extract securely and store in the freezer. Record the final volume in the extraction log.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batch requirements, these must be followed instead.

Each extraction batch (up to 20 samples) must contain a method blank, an LCS, and either an unspiked background sample, and MS/MSD, or an LCS/LCSD.

QC limits for surrogates, LCS/LCSD, and MS/MSD are established through statistical analysis of historical data. The limits are evaluated every 6 months and updated as needed. The limits are maintained in the LIMS for the relevant analysis numbers.

If the batch contains only field or equipment blank samples, the LCS/LCSD QC pairing should be used.

End of document

Version history

Version	Approval	Revision information
14	30.SEP.2016	

eurofins	Always check on-line for validity **Separatory Funnel Extraction by Method 3510C for BNAs by 8270 SIM in Wastewater**	Level: **Work Instruction**
Document number: **T-OE-SVOA-WI10931**		
Old Reference: **1-P-QM-WI -9015121**		
Version: **8**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **30-SEP-2016**	Document users: **5_EUUSLA_GC/MS Semivolatiles_Manager, 5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Semivolatile Waters, 6_EUUSLA_GC/MS Semivolatiles_Analysts**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID
Analysis DOD - 11012, 10465, 10466, 10470, 10471, 11912

Revision Log

Revision: 8	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects current analysis numbers	Added analysis numbers 14243, 14244, 14245
Apparatus and Equipment	Reflects current practice	Added Automated Water Extraction Bench, Rapid-Vap Evaporator, Rapid-Vap Concentrator Tubes
Procedure G.1. and G.2.	Reflects current practice	Added spike and surrogate details for new analysis numbers
Procedure L.-X.	Reflects current practice	Separated 1L extraction from 250-mL extraction and added new procedure for 250-mL extraction.

Revision: 7		**Effective Date:** Jul 1, 2016
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Apparatus and Equipment	Reflects current equipment in use	Changed balance accuracy to 0.1g Added Teflon separatory funnel and pH paper-wide range
Procedure H. & I.	Reflects current process	Reordered steps
Procedure M.	Reflects current process	Added detail on steps for handling emulsions
Procedure N.	Enhancement	Added height of sodium sulfate column in inches for both 1000mL and 250 mL extractions
Procedure O.	Reflects current process	Added step to rinse sodium sulfate column after first solvent drain.
Procedure P & Q	Reflects correct procedure references	Revised procedure step references
Procedure Q	Reflects current process	Added approximate amount of drops of sulfuric acid to acidify the sample. Added pH check and documentation on batch log
Procedure R.	Reflects current process	Changed amount of methylene chloride to 15 mL.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3510C, Rev 3, December 1996.
2. *Chemical Hygiene Plan,* current version.

Cross Reference

Document	Document Title
Analysis #8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971	Semivolatiles by Methods 8270C/D SIM
Analysis #10242, 10262, 11305, 11597	Determination of Parent and Alkyl Substituted Polynuclear Aromatic Hydrocarbons (PAHs), Alkanes and Geochemical Biomarkers by Gas Chromatography/Mass Spectrometry (GC/MS-SIM)
1-P-QM-PRO-9015452	Semivolatile Spiking and Calibration Standards
1-P-QM-PRO-9015475	Glassware Cleaning for Organic Extractions
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Scope

This method is for the extraction of semivolatile organic compounds at low ppb to low ppm levels in water and wastewater matrices that are not prone to emulsions. High levels of organic compounds and/or extreme alkalinity or acidity in the sample often interfere with normal detection limits.

Basic Principles

A portion of sample is placed into a separatory funnel. Surrogate standards are added to each sample to monitor recovery. The pH of the sample is adjusted to >11 and the sample is serially extracted with methylene chloride. The pH of the sample is then adjusted to <2 and the sample is extracted with methylene chloride. The solvent fractions are combined, and the extract is dried and concentrated.

Procedural changes to use a reduced sample aliquot while maintaining the default reporting limits are permitted as long as the reagent aliquots are reduced proportionally and a quad study is performed and on file.

Reference Modifications

Surrogate and matrix spiking solutions are not added before the transfer to the separatory funnel for several reasons:

1. Samples must be poured from the amber bottles to determine the matrix and volume of sample to use for each extraction.

2. Many sample bottles have no headspace and there is no room to add surrogate to the sample in the bottle.

3. Due to the volume of samples extracted, a separate graduated cylinder for each sample is unrealistic.

4. To maintain consistency with all extractions, no samples are spiked in the bottle or graduated cylinders.

Interferences

Impurities in solvents, reagents, glassware, or other hardware used in sample processing lead to interferences with the method. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of sample to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the technicians training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the extraction and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, or one blind sample.

Sample Collection, Preservation and Handling

Samples are collected in glass bottles with PTFE-lined lids and stored at 0° to 6°C, not frozen, prior to extraction. Samples must be extracted within 7 days of sample collection. The extract is stored in an amber autosampler vial in the freezer ≤-10 °C for up to 40 days prior to analysis.

Apparatus and Equipment

1. 2-L separatory funnel for extracting organic components from an aqueous matrix
2. Kuderna-Danish (K-D) assembly with appropriate ampule for extracting the solvent used during the extraction
3. Water bath – VWR/LLI Model #1127 or equivalent
4. Graduated cylinders – Class A, assorted sizes
5. Pipettes – Class A, assorted sizes
6. Pipettes – Disposable
7. Solvent pumps – Brinkman, adjustable
8. Balance – Capable of weighing to 0. 1 g
9. Automatic shaker – Capable of holding 2 L separatory funnels
10. Centrifuge – Beckman GS-6 or equivalent
11. Sodium sulfate columns

12. Micro-snyder columns

13. Wash bottles – Teflon

14. Amber autosampler vials

15. Teflon boiling chips

16. Teflon separatory funnel

17. pH paper – Wide range

18. Automated Water Extraction Bench – capable of holding separatory funnels for 250−mL extractions

19. Rapid-Vap Evaporators – for concentration of 250−mL extractions

20. Rapid-Vap Evaporator Tubes – Class A

Reagents and Standards

1. Methylene chloride (CH_2Cl_2) – Pesticide grade or equivalent. Store at room temperature for up to one year.

2. 10N Sodium hydroxide (NaOH) – Lab chem or equivalent. Store at room temperature for up to one year.

3. Sulfuric acid – ACS grade or equivalent, concentrated. Store at room temperature for up to 1 year.

4. Sodium sulfate (Na2SO4) – Granular anhydrous reagent grade or equivalent. Bake at approximately 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

5. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical Analysis #8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971, 14243, 14244, 14245 and Analysis #10242, 10262, 11305, 11597.

Preparation of Glassware

See 1-P-QM-PRO-9015475 (SOP-OE-001).

Calibration

Not applicable to this procedure.

Procedure

A. Determine the volume of sample to be used for each extraction. Ideally, one full bottle is used. (Typically this is 1L for routine extraction or 250−mL for mini-extractions).

1. 1 Liter analysis numbers: 8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971, 10242, 10262, 11305, 11597.

2. 250 mL analysis numbers: 14243, 14244, 14245

B. If the sample bottle is a 1-L bottle (or 250−mL bottle) and is not a quality control sample [Background (BG), matrix spike (MS), matrix spike duplicate (MSD)], mark the water level on the outside of the sample bottle in order to later determine the sample volume.

1. Shake the bottle vigorously.

2. Pour the sample into a separatory funnel.

3. Record any comments about the sample on the extraction sheet.

C. If the sample bottle is larger than 1−L (or 250 mL) or the sample is a quality control sample, exactly 1 L (or 250 mL) of sample is extracted.

1. Shake the sample bottle vigorously.

2. Use a clean graduated cylinder to measure the necessary volume of sample and pour it into a separatory funnel.

3. Record the sample volume and any comments about the sample on the extraction log.

4. Use a wash bottle to rinse the graduated cylinder with methylene chloride and add the rinseate to the separatory funnel.

D. **NOTE:** Reduced volume of sample is only used with prior approval of the client or if the matrix of the sample does not allow the extraction to proceed using full volume. The sample must then be diluted to 1 L (or 250 mL).

E. The method blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) (if applicable) are prepared using 1 L (or 250 mL for mini−extractions) of non−deionized reagent water (tap water) measured into the separatory funnel.

F. The background, MS, and MSD are prepared on three separate aliquots of a field sample.

G. Surrogates and Spiking Solutions

1. Surrogates

a. Analysis 0039, 8357, 10010, 10262, 10137, 11305, and 11913 - Add 1.0 mL BNA surrogate to all samples, blanks, LCS, LCSD (if applicable), MS, and MSD..

b. Analysis 14243, 14244, and 14245 – Add 1.0 mL Mini BNA Surrogate to all samples, blanks, LCS, LCSD (if applicable), MS, and MSD.

NOTE: Be certain the standard drips directly into the sample without touching the glass side of the separatory funnel to avoid poor recoveries.

2. Spiking solutions

a. Analysis 0039, 8357, 10137 and 11913 - Add 1.0 mL of SIM spiking solution to the LCS, LCSD if applicable, MS, and MSD.

b. Analysis 10010 – Add 1.0 mL of SIM spiking solution to the LCS, LCSD if applicable MS, and MSD. Spike a separate LCS, LCSD with 1.0 mL of Benzenethiol spiking solution

c Analysis 10262 – Add 1.0 mL of Alkyl PAH spike to the LCS, LCSD, if applicable MS, and MSD.

d. Analysis 11305 – Add 1.0 mL of DPnB spiking solution to LCS, LCSD if applicable, MS and MSD.

e. Analysis 14243, 14244, 14245 – Add 1.0 mL of Mini SIM spiking solution.

f. If the sample requires additional semivolatile compounds, 1.0 mL of 1 ppm spike (or 0.25 mL for 250−mL extractions) of this compound is added at this time.

g. See analysis SOPs for spike details.

3. See 1−P−QM−PRO−9015452 (SOP-EX-001) for preparation of spikes and standards.

4. See 1−P−QM−PRO−9015490 (SOP−OE−017) for storage and handling of spikes.

H. Determine the volume of sample used for extraction:

1. If the original sample bottle is empty:

a. Use a solvent pump to measure 60 mL of methylene chloride (15 mL for 250−mL extractions) and rinse the sample bottle by capping and inverting several times prior to adding the methylene chloride to the separatory funnel.

b. Transfer the solvent to the separatory funnel.

c. Fill the bottle to the marked level with non−deionized reagent water (tap water) and pour into a graduated cylinder to determine the initial volume.

2. Alternatively, tare the empty sample bottle on the balance.

a. Fill the bottle with non−deionized reagent water (tap water) to the marked line and place the bottle back onto the balance.

b. Round the weight to a whole number. This is the volume of sample used for extraction.

3. If the sample container is not empty, use a solvent pump to measure 60 mL of methylene chloride (15 mL for 250−mL extractions) and add the solvent directly to the separatory funnel.

I. Add 10N sodium hydroxide to each sample, Method Blank, LCS/LCSD to adjust the pH to >11.

NOTE: If samples are entered for 8357, 10262, 11305, or 11913 proceed to Procedure R.

J. Cap the funnel, invert, and vent immediately.

K. Handshake and vent frequently until the pressure is stable.

L. For analyses 8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971, 10242, 10262, 11305, 11597: Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

For analyses 14243, 14244, 14245: Separatory funnels remain on the Automated Water Extraction Bench. Press the green “start” button to lower the hood and activate the Automated Water Extraction Bench. The bench will tumble for 2 minutes. The sash will rise after the samples have been tumbled.

NOTE: Shaker speeds vary greatly between instruments; the proper setting is marked on each.

M. For analyses 8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971, 10242, 10262, 11305, 11597: Place the separatory funnel on the rack and allow it to sit undisturbed for approximately 10 minutes. For analyses 14243, 14244, 14245, the separatory funnel will sit undisturbed on the Automated Water Extraction Bench.

The time required for extracts to set undisturbed is based upon visual confirmation that the layers are adequately separated. Additional time may be necessary for samples with unusual high density (i.e. high salt content).

If an emulsion forms and is >1/3 of the volume of the solvent layer, mechanical techniques such as pouring the emulsion into a Teflon separatory funnel and centrifugation (2500 rpm for 2 minutes) must be employed to complete the separation.

N. For analyses 8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971, 10242, 10262, 11305, 11597: Drain the solvent layer into a metal beaker and pour it through approximately 10 cm (~4 inches) of sodium sulfate into a K-D apparatus containing Teflon™ boiling chips. For analysis 14243, 14244, 14245, drain the solvent layer directly into the sodium sulfate column containing approximately 5 cm (~2 inches) of sodium sulfate. This drains into a Rapid-Vap Evaporator tube.

O. Rinse sodium sulfate column with approximately 20 mL of methylene chloride (approximately 15 mL of methylene chloride for 250−mL extractions).

P. Use a solvent pump to add 60 mL of methylene chloride (15 mL for 250−mL extractions) to the separatory funnel and repeat Procedure steps J through N, venting only as necessary.

Q. Again, use a solvent pump to add 60 mL of methylene chloride (15 mL for 250−mL extractions) to the separatory funnel and repeat Procedure steps J through N, venting only as necessary. The acid extract is also added to this KD.

R. Add approximately 10-13 drops of sulfuric acid with a disposable pipette to adjust the sample pH to <2. Check pH using wide range pH paper and record on batch log. Using three 60−mL aliquots of methylene chloride (15 mL for 250−mL extractions), serially extract the sample as described in Procedure J. through N.

For analyses 8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971, 10242, 10262, 11305, 11597: Rinse metal beaker with approximately 20 mL of methylene chloride. Pour the solvent from the metal beaker into the sodium sulfate column. For analyses 14243, 14244, 14245, drain the solvent layer directly into the sodium sulfate column.

S. Use a wash bottle to rinse the sodium sulfate column with approximately 20 mL of methylene chloride (15 mL for 250−mL extractions). Use a hand held bulb to squeeze any excess methylene chloride through the sodium sulfate column.

T. For 1−Liter extractions

1. Attach a 3-ball Snyder column to the K-D, wet with solvent, and concentrate the extract to approximately 1−mL on a steam bath at 80º to 90ºC. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. Adjust the vertical position of the apparatus and the water temperature as needed to complete the concentration in 10 to 20 min

2. Allow the sample to cool for 10 minutes.

3. Microsnyder the sample to slightly below 1 mL.

4. Bring the combined extract to 1.0 mL using methylene chloride.

5. Determine the final volume by placing the extract into an amber autosampler vial and comparing the level in the vial to a reference vial containing the exact targeted final volume. Add methylene chloride to the extract with a disposable pipette until exactly the same level is in both vials.

If too much solvent is added to the sample vial, remove the extract from the vial and concentrate it by microsnydering to slightly less than the targeted final volume and rebottle.

a. Cap securely and store in the freezer.

b. Record the final volume on the extraction log.

U. For 250−mL Extractions

1. Place the evaporator tube containing the solvent extract into the pre-heated (approximately 80°C) Rapid-Vap Evaporator. Set the timer for 15 minutes and speed at 75%(the speed is the rotation within the unit while the sample is evaporating). Confirm that the nitrogen is set at 15 psi (this is adjusted on the regulator attached to the nitrogen line). Allow the samples to concentrate to around 0.5 mL. Bring to final volume 1 mL in the Class A Concentrator Tube by rinsing with Methylene Chloride.

NOTE: The temperature, timer, and speed settings can be adjusted as necessary to accommodate difficult sample matrices, etc.

2. Mix the sample thoroughly in the Concentrator Tube and transfer the extract to an amber autosampler vial. Cap the extract securely and store in the freezer. Record the final volume on the extraction log.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. For each batch of samples extracted, a blank, an LCS, an MS, and MSD must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead. Also, if the batch contains only field or equipment blank samples, the LCS/LCSD QC pairing must be used.

If any client, state, or agency has more stringent QC or batch requirements, these must be followed instead.

End of document

Version history

Version	Approval	Revision information
8	30.SEP.2016	

An error has happened on file: /D4Doc/formularer/2016/09/Quality Document # 1-P-QM-WI -9015121_7_1101210465.doc
Errortext: The remote server returned an error: (404) Not Found.

eurofins	Always check on-line for validity **Determination of Volatile Target compounds and Gasoline Range Organics (GRO) by GC/MS in Soils and Solids by Method 8260C**	Level: **Work Instruction**
Document number: **T-VOA-WI8236**		
Old Reference: **1-P-QM-WI-9013077**		
Version: **4**		Organisation level: **5-Sub-BU**
Approved by: **UCSS** Effective Date **24-JUN-2016**	Document users: **6_EUUSLA_GC/MS Volatiles_GC/MS Auditors/Verifiers, 6_EUUSLA_GC/MS Volatiles_Management, 6_EUUSLA_GC/MS Volatiles_SW846 8260B/C Soil**	Responsible: **5_EUUSLA_GC/MS Volatiles_Manager**

LIMS ID
Analysis 11995

Revision Log

Revision: 4		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Reference	Current Practice	Added 5030C
Reference 2. to 6.	Update	Changed to reflect the LIMS format
Scope	Requirement Current Practice	Added verbiage concerning MDL/LOQs in the LIMS Added verbiage concerning TSC sheets

Revision: 4		**Effective Date:** This version
Basic Principles	Generalization	Removed specific scan references and replaced with corresponding SOP reference
Reagents and Standards B.3	Generalization	Removed reference to specific autosampler model
Reagents and Standards B.3.c	Requirement	Added preparer name and storage conditions to standard label requirements
Reagents and Standards B.4	Clarification	Adjusted wording for clarity
Calibration A	Unnecessary information	Removed SIM parameters since addressed in separate SOP
Calibration C.1	Clarification	Removed specific number of levels reference concerning high level surrogates
Calibration C.4	Generalization	Removed reference to specific autosampler model
Procedure C	Generalization	Removed specific scan references and replaced with corresponding SOP reference
Calculations	Consistency	Moved equations from Procedure to Calculations section
Quality Assurance/ Quality Control	Correction	Replaced “Tables” with “Figures” in regard to QC TSC sheets
Attachment I Basic Principles	Current Practice	Added C5 marker
Attachment I Calibration A.1	Update	Removed HL specific calibration levels since both HL and LL calibration levels are now the same
Attachment I Entire Attachment	Update	Changed Figure reference to Figure 6 only
Appendix	No longer applicable	Removed Figure 7
Appendix	Update	Replaced Figures 1-6 with most current version

Revision: 3		**Effective Date:** May 19, 2014
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Title	EtQ requirement	Shortened document title to read as follows “**Determination of Volatile Target Compounds and Gasoline Range Organics (GRO) by GC/MS in Soils and Solids by Method 8260C**”
Reference	Current practice	Added Method 5030B
Sample Collection, Preservation and Handling	Current requirement	Updated temperature range
Reagents and Standards A.2	Current practice	Added “or equivalent” to methanol description

Reference

1. Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), SW-846 Method 8260C, August 2006.

2. Determinative Chromatographic Separations, SW-846 Method 8000C, March 2003.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 5030B, December 1996.

4. Test Methods for Evaluating Solid Wastes, SW-846 Method 5030C, May 2003.

5. Test Methods for Evaluating Solid Wastes, SW-846 Method 5035, Revision 0, December 1996.

6. Test Methods for Evaluating Solid Waste, SW-846, Method 5035A, July 2002.

7. *otal Petroleum Hydrocarbons Analysis-Gasoline Method*, California Department of Health Services, LUFT Task Force.

8. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
1-P-QM-WI-9015069	GC/MS - Bulk Solid Matrix Sample Preparation
1-P-QM-WI-9015193	Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035
1-P-QM-PRO-9015467	GC and GC/MS Instrumentation Maintenance
1-P-QM-PRO-9015469	GC/MS Volatile Standards Traceability
1-P-QM-PRO-9015470	Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles
1-P-QM-PRO-9015471	GC/MS Volatiles Audit Process
1-P-QM-QMA-9015390	Demonstrations of Capability
1-P-QM-QMA-9017309	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is suitable for the determination of target compounds listed and maintained in the Laboratory Information Management System (LIMS) for soil and solid matrices. Samples consist of soils/solids collected or submitted in bulk in glass containers with Teflon™-lined screw−caps or in field core sampling/storage containers (i.e., EnCore™ or equivalent). Associated MDLs/LOQs are listed in the LIMS under the analysis numbers and/or Project Information lists. Theoretical Standard Calibration (TSC) Sheets are included in the Appendix (Figures 1-6). These TSC sheets are to serve as examples only and may not reflect most current version in use. Non−target volatile compounds in the sample can be tentatively identified (TIC) using a mass spectral reference library comparison. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by purge and trap GC/MS methodologies and skilled in mass spectral interpretation. Using this method, the TICs are quantitated with an estimated concentration.

Compounds other than those listed in the LIMS for this group of master scans may be analyzed using USEPA SW−846 Method 8260C. Selected Ion Monitoring (SIM) parameters can be used to detect, identify and quantitate volatile organic compounds in methanol extracts of soils/solids, if lower quantitation limits are required (project− or client−specified) and/or matrix interferences are anticipated.

Attachment I describes the proper analysis procedure for Gasoline Range Organics in soils and solids. Due to poor purging efficiency or poor chromatography, some analytes require calibration at higher levels and/or higher practical quantitation limits (PQLs). Any additional compounds must be added to the theoretical standard concentrations (TSC) sheet. Standards containing additional analytes must be prepared as described in the Standards section of this document. Both secondary stock solutions and matrix spike solutions must be prepared for use in analyzing additional compounds.

Basic Principles

The soil sample is prepared according to analysis 1-P-QM-WI-9015069 and 1-P-QM-WI-9015193 for 8260C. The sample is purged with an inert gas and the effluent gas passed through a sorbent trap where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and backflushed onto the head of a gas chromatographic column held at the appropriate initial temperature for the column in use. The gas chromatographic column is temperature programmed to separate the volatile compounds, which are subsequently detected and identified using mass spectrometric techniques.

When a compound reaches the MS, it is bombarded by high energy electrons (70 eV). This causes the compound to fragment and form ions. The positive ions are focused into a quadrupole mass analyzer, where the ions are separated according to their mass/charge ratios during rapid repetitive scans. These ions are then amplified and detected with an electron multiplier.

The resulting time/intensity/mass spectra data are stored and processed by a computer. Target compounds are identified on the basis of relative retention times and mass spectral matches to standards, which are injected every 12 hours on the same system. The internal standard method is used for quantitation.

Interferences

Contaminant sources are volatile compounds in the laboratory environment, impurities in the inert purging gas, carryover from samples containing high concentrations of volatile organic compounds and dirty glassware. The analyst must demonstrate that the system is free from interferences (by producing acceptable method blank data) before analyzing a batch of samples. Matrix effects from heavily contaminated soils and solids can interfere with the internal standard responses, target analytes and surrogate recoveries, thereby hindering accurate quantitation. See Section 4.0 of SW−846 Method 8260C for further discussion.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity of each reagent used has not been precisely determined. However, each reagent must be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves when working directly with reagents. Refer to the *Chemical Hygiene Plan* for specific details.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability.

Education Requirement: A 4–year Baccalaureate Degree from an accredited College or University in one of the physical sciences and/or 1 – 3 years of relevant gas chromatography experience.

Analysts must be trained in the proper method of volatile organic sample preparation and analysis as determined by the supervisor(s). All training and education relating to volatile organic sample preparation and analysis must be documented by each analyst in their training record. Specifically, each new chemist must train with an experienced chemist for the first 12 weeks depending on the individual and their previous experience. The first 12 weeks are spent working one-on-one with the trainer. This time may be less if the new chemist has prior relevant experience in GC/MS and analytical chemistry background.

During the training period, the new chemist must learn daily maintenance, calibration techniques, data and library search review, and forms generation. They are also required to read all relevant SOPs and EPA methods.

To measure the proficiency of each chemist, several checks have been established. The first is the ability to successfully calibrate. The chemist analyzes a series of at least five calibration standards and performs the calibration routine. Secondly, each analyst must perform a Demonstration of Capability (DOC). Refer to 1-P-QM-QMA-9015390 for specific requirements. Demonstration of Capability is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

The samples must be stored in a refrigerator at 0°C to 6°C, not frozen. Soils are collected/submitted in bulk in glass containers with Teflon™-lined screw-caps or in field core sampling/storage containers (i.e., EnCore™ or equivalent). Samples are preserved in methanol and either frozen reagent water or Sodium Bisulfate. All samples must be analyzed within 14 days of collection.

Apparatus and Equipment

1. Gastight micro syringes – 10-µL and larger

2. 5-mL gastight syringes

3. Analytical balance, capable of accurately weighing ±0.0001 g

4. Top-loading balance capable of weighing ±0.01 g

5. Glassware

   a. Class A Volumetric flasks with ground-glass stopper

   b. Vials, 1.5-mL, 15-mL, and 40-mL screw cap, with Teflon/silicone septa

   c. Mininert vials, 1-mL, 2-mL, and 5-mL

6. Purge and trap device – Consisting of the sample purger, the trap, and desorber; the OI Analytical 4560, OI Analytical 4660, or equivalent meets the requirements of this method. The purging chamber must have the purge gas passing through the sample as finely divided bubbles and minimize the headspace between the sample and the trap to <15 mL.

7. Autosampler – OI Analytical 4551, Archon, or equivalent meets the requirements of this method. The Archon has the capability to purge solid samples directly in the 40-mL vial (needed for samples prepared by analysis 8389).

8. Spiker units (optional) – OI Analytical Model 4551 SIM/Spiker or equivalent. One or two automated syringe spikers can be added to the OI Analytical Model 4551 autosampler to automatically introduce 10 µL of internal standard (ISTD), surrogate standard, and/or matrix spiking solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of appropriate standards.

9. GC/MS system – The HP 5890GC/5972 MSD, HP 6890GC/5973MSD, HP 6890GC/5975MSD, and Shimadzu GC/MS QP5000 meet the requirements for this method.

10. Data System/Computer/Software– this is interfaced to the GC/MS system, which continuously acquires and stores data during the analysis, and can process/reduce data to generate the appropriate

forms and supporting data. The software used for acquisition is HP Chemstation®, and data reduction is accomplished using Target® software.

11. GC Columns

a. olumn 1 – 30M × 0.25 mm ID DB624 capillary column with a 1.4−µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

b. Column 2 – 20M × 0.18 mm ID DB624 capillary column with a 1.0−µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

Different sampling/analysis combinations are used based on how the sample was collected, expected level of VOCs in the sample, possible matrix interferences, list of target compounds, whether TICs were requested, the required quantitation limits and the type of equipment/instrumentation available.

NOTE: Refer to 1-P-QM-PRO-9015467 for instrumentation maintenance and troubleshooting.

Reagents and Standards

A. Reagents

1. Reagent water is defined as water in which an interferant is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory must meet these criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Purge and Trap grade Methanol or equivalent

3. Sodium bisulfate – ACS grade

4. Sand- Ottawa Standard

B. Standards

See 1-P-QM-PRO-9015469 for standards traceability.

1. Stock standard solutions – Stock solutions must be prepared in methanol. Standards are prepared from ampulated and neat compounds obtained from suppliers that indicate the purity of the compound. No correction for purity is made if the purity is listed as ≥96%. Pre-made solutions can be used if the supplier documents the concentrations of the solutions. All ampulated standards are stored at −10 to −15°C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided.

NOTE: For most of the target compounds, the stock standard solutions are purchased from a vendor as custom mixes (V for calibration and Q for separate source quality control). The internal and surrogate

standards are purchased from a vendor, as well as the target compounds that are gases at room temperature. These gaseous standards have a 1−week expiration date, starting from the date they are opened.

2. Surrogate stock standard solution (for high level soils) – a 2500 µg/mL stock standard solution of dibromofluoromethane, toluene-d8, 4−bromofluorobenzene, and 1,2−dichloroethane-d4 is prepared in methanol by a commercial supplier

3. 8260A Internal standard spiking solution (8260IS) – a 2500 µg/mL stock standard solution of fluorobenzene, chlorobenzene−d5, 1,4−dichlorobenzene−d4, and 12500 µg/mL deuterated tertiary butyl alcohol (tBA−d10) is prepared in methanol by a commercial supplier. Deuterated tertiary butyl alcohol (tBA−d10) is used sometimes as an auxiliary ISTD. If an autosampler is used, dilute the stock to 250 µg/mL (1250 µg/mL for tBA-d10) in methanol. This is assuming a 1−µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained (50 µg/kg or 1 µg/kg for SIM scan).

To prepare stock standards from neat compounds:

a. Place about 9.8 mL methanol or an equivalent solvent into a tared 10.0−mL glass-stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

b. Add the liquids using a syringe or pipette by adding 2 or more drops of the assayed material to the flask, being careful that no drop hits the side of the flask. Reweigh the flask, record/note the amount, dilute to volume, stopper, and mix by inverting the flask at least 3 times. Calculate the concentration of the standard.

c. The stock standard solutions are stored in Teflon−sealed screw−capped vials at −10 to −15°C. The compound name, concentration, date prepared, expiration date, preparer name and storage conditions must appear on the bottle.

d. Replace in house prepared stock standard solutions every 6 months.

4. Secondary dilution standards – Using the stock standard solutions, prepare secondary stock solutions in methanol containing the desired compounds. These standards are prepared by calculating the volume of each stock standard required producing a given volume of a mixed working standard with a known concentration of each analyte. When custom mixes are used, these may be diluted down individually, or combined together with other mixes. The working standard is tested according to 1-P-QM-PRO-9015469. The verified working standard is poured into Teflon-lined screw−capped GC vials or mininert vials and stored at −10 to −15°C. A designator indicating the standard, month, and preparation date must be on the standard vials. The designator and all data pertaining to the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every 6 months unless otherwise indicated.

a. 1,4-Bromofluorobenzene (BFB) standard – Prepare a 50−µg/mL solution of BFB in methanol by diluting the stock standard (prepared from neat material) with methanol to a final volume of 100 mL. The volume of stock standard used varies, depending on the actual stock concentration.

b. Surrogate standard spiking solution (high level only) – Prepare the surrogate standard spiking solution from the stock standard solutions at a concentration of 25 or 250 µg/mL in methanol.

c. Internal standard (IS) solution (high level only) – fluorobenzene, chlorobenzene−d5, 1,4−dichlorobenzene−d4, and tert-Butyl Alcohol-d10. One mL of 8260IS is diluted with methanol to a total volume of 10 mL to give a final concentration of 250 µg/mL (1250 µg/mL for tBA-d10). This is assuming a 1-µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained.

d. IS/SS spiking solution – Dilute 1 mL of 8260IS and 1 mL of 8260SS with methanol to 10−mL final volume (resulting in a concentration of 250 µg/mL, 1250 µg/mL for tBA-d10). This is assuming a 1−µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained.

e. Calibration spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. Suggested calibration levels are 4, 10, 20, 50, 100, and 300. Replace the calibration spiking solution every month.

f. Matrix spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. These solutions serve as both the matrix spiking solution and the laboratory control sample solutions. Matrix spikes also serve as duplicates; therefore, two aliquots of the same sample need to be spiked for analysis with these solutions. Replace the matrix spiking solution every month.

g. Store all standard solutions at −10 to −15°C.

Calibrations

A. Instrument Conditions:

1. The purge and trap device must have the trap conditioned for at least 10 minutes at 180° to 220°C at a flow rate of 20 to 60 mL/min prior to initial use.

2. An example of purge and trap conditions are listed below:

Purge gas	Helium
Purge flow	35 – 40 mL/min
Purge temperature	40°C for low level soils and ambient temp. for medium/high level soils
Purge time	11 min

Desorb temperature	190°-220°C
Desorb time	0.5 to 4 minutes **
Bake temperature	180°-220°C
Bake time	8 min (±3 min)

** - Range as suggested by the purge and trap instrument manufacturer

NOTE: Purge and trap conditions may be changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument may be found in the appropriate instrument maintenance log.

3. The suggested gas chromatographic operating conditions are listed in the table below, depending on the column used:

	Column 1	Column 2
Column liquid phase	DB-624	DB-624
Carrier gas	Helium	Helium
Carrier gas flow	.8 mL/min	.6 mL/min
Make-up gas flow	None	None
Initial temperature	45°C	45°C
Initial hold time	4.5 min	2.5 min
Temperature ramp	12°/min to 100°C then 25°/min to 240°C	12°/min to 100°C then 25°/min to 235°C
Final temperature	240°C	235°C
Final hold time	None	.02 min

4. Recommended mass spectrometer (MS) operating conditions:

Ions	Positive
Electron energy	70 volts
Mass range	35 – 300 amu

H-P systems Scan time:

Number A/D Samples	2^2 (4)
Integration Time/Sample	50 microsec
Total Scan Time	0.6 sec

B. Tuning:

Tune the GC/MS system to meet the criteria in Table 1 following a 50−ng injection of BFB. The chromatographic conditions must be the same as those under which the samples are analyzed except

that the temperature ramp may be increased and the initial temperature and flow rate may be different. The BFB tune must be verified every 12 hours.

The tune must be evaluated by taking the average of the three scans across the BFB peak apex with a background subtraction of a scan within 20 scans prior to the start of the BFB peak.

NOTE: All standards, samples, and associated quality control samples must be analyzed with the same mass spectrometer parameters as those used to obtain a successful tune.

C. Initial Calibration:

1. Perform the initial calibration by analyzing at least six distinct levels of analyte concentration. For high level soils, the surrogates are also analyzed at each distinct level. Response factors for each analyte are determined from these levels. Six levels are required if second order regression fits are used. Refer to Figure 1 for the preparation of calibration standards. The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of the analyte concentration.

NOTE: 5 levels are required by the method.

2. A method detection limit (MDL) standard must be analyzed with each initial calibration. This standard is prepared at or near the departmental MDL and is not to be included in the calibration curve. All compounds must be detected in the MDL standard.

3. For medium/high level soils, methanol is added to all calibration standards. For low−level soils prepared according to EPA Method 5035, 1 scoop of sodium bisulfate is added to all calibration standards.

4. When using an autosampler, blanks and standards are prepared and poured into 40−mL vials with Teflon−lined septa. For the high−level method, 5 mL is withdrawn from the vial and transferred to the sparge vessel along with the appropriate amount of the internal standard spiking solution. For the low−level method, the autosampler transfers 5 mL of reagent water along with the appropriate amount of IS/SS spiking solution to the 40−mL vial.

5. Purge and desorb according to Calibration 2.

6. Collect GC/MS data until the end of the GC run.

7. Empty and rinse the purging chamber at least twice with reagent water prior to loading another sample into the vessel, to minimize the possibility of carryover contamination.

8. Each level is analyzed as described above. Next, tabulate the area response of the characteristic ions (Table 2) against concentration for each analyte, surrogate standard, and internal standard and calculate relative response factors (RRF) for each compound (see Calculation section). The following table describes the guidelines for an acceptable initial calibration:

Frequency	Acceptance Criteria	Corrective Action
Initially and then when analytes in the daily calibration standard fail criteria.	1. % RSD of ≤20% is required for all analytes. 10% of the analytes may fail this criteria. 2. All compounds of interest must be detected in the MDL standard. 3. The relative retention times of the target compounds must agree within 0.06 relative retention time (RRT) units. The exception would be in the case of system maintenance. 4. Minimum response factors must be met for select compounds. See Table 3.	1. Any target analyte with a %RSD of ≤20% must use the average RRF for quantitation. For any analyte in which the %RSD >20%, a first-degree linear regression can be used (providing that the correlation coefficient [CC] is ≥0.99). A quadratic fit ** (using 6 stds) can also be used (provided the coefficient of determination [CD] is ≥0.99). If the linear fit and quadratic fit pass the criteria for any given analyte, then use the line/curve with the smallest positive y-intercept. If the y−intercept quantifies to be greater than the LOQ, consult your supervisor immediately or recalibrate. If CC or CD is <0.99, recalibrate. Supervisory approval is required for exceptions to these guidelines. If >10% target analytes fail, recalibration is required. 2. If a compound is not detected in the MDL standard, then report to the level of the lowest standard detected. 3,4. Perform system maintenance and recalibrate.

**Consult USEPA method 8000B for non-linear curve fitting techniques/guidelines

NOTE: If a linear fit is used for a compound, the lowest calibration standard point must be recalculated against the curve. The recalculated concentration must be within ± 30% of the standard's true concentration. If this criterion is not met, notify a supervisor so that an alternate LOQ can be evaluated.

D. Initial Calibration Verification:

Following the calibration, an Initial Calibration Verification (ICV) standard must be run. The ICV is prepared according to the TSC sheet in Figure 5. The ICV acts as a second source standard to check against the initial calibration. All analytes must meet ICV acceptance windows of 70%-130%. If the ICV does not meet the aforementioned criteria, a second ICV is analyzed before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

E. Continuing Calibration:

Continuing Calibration Verification (CCV) – The CCV is performed by analyzing a 50-ppb calibration standard in subsequent tune periods after an initial calibration. The CCV is considered valid when the criteria listed below are met:

Frequency	Acceptance Criteria	Corrective Action
Every 12 hours.	1. % Drift of ≤20% is required for all analytes. 20% of analytes may fail this criteria if not detected in proceeding samples. 2. The relative retention times (RRT) of the target compounds must agree within 0.06 RRT units. The exception would be in the case of system maintenance. 3. The extracted ion current profile (EICP) area for each internal standard must fall within the window of –50 % to +100 % from the mid-level standard area produced during the last initial calibration. 4. Minimum response factors must be met for select compounds. See Table 3.	1. - 4. In the event that the continuing calibration verification (CCV) standard fails any of these criteria, sample analysis must be suspended and the CCV must be re-analyzed. If the re-analysis fails any of the criteria then adjustments are to be made to the analytical system to return it to its original condition, followed by the analyses of 2 consecutive CCVs at the same level that failed. If both CCVs pass the criteria, then sample analysis can continue. Otherwise, the system must be recalibrated and the samples reanalyzed, or the data can be qualified.

Calibration Calculations

1. Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Characteristic ion area for the compound being measured

A(is) = Characteristic ion area for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression equations:

1st Order (linear) regression: $Y = Mx + B$

2nd order (quadratic) regression: $Y = Cx2 + Mx + B$

Where:

x = Area(Std) /Area(Istd)

Y = Conc.(Std)/Conc.(Istd)

M = 1st degree slope

C = 2nd degree slope

B = Y-intercept

3. Percent relative standard deviation (%RSD):

$$\%\ RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

4. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

CI = Measured concentration using selected quantification method

Procedure

A. Method Blank:

Analyze the method blank as described above for the initial calibration standards. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than the reporting limit for the associated samples. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met.

B. Laboratory Control Sample/ Duplicate and Matrix Spike/Duplicate: Refer to table in QA/QC section for specific requirements.

C. Qualitative Analysis:

Sample analysis for soil, solids, and nonaqueous matrices proceeds as described in the Calibration section. The aqueous matrix is replaced by the soil sample, which is prepared according to 1-P-QM-WI-9015069 and 1-P-QM-WI-9015193. A compound is identified by comparison of the following parameters with those of a standard of this target compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The compound relative retention time must compare within ±0.06 RRT units of the RRT of the standard.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst discretion is used to determine compound identification. (Example: for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

5. The above criteria apply to hits greater than or equal to the LOQ. For hits between the MDL and the LOQ, both the above criteria and analyst discretion are used to determine compound identification.

6. The analyst must account for peaks that are greater than 10% relative intensity in the sample mass spectrum, but not present in the standard mass spectrum. Also, if a compound fails any of the criteria

listed above but in the judgment of the mass spectral interpretation specialist is a correct identification, the identification is used and the quantitation of the peak is performed.

The primary and secondary ions can be found in Table 2.

D. Quantitative Analysis:

Once a compound has been identified, quantitation of identified priority pollutant compounds is performed using the equations listed in the Calculations section of this SOP. The primary ions listed in Table 2 are used for quantitation. A secondary ion may be used if there is interference with the primary ion. All calculations must report concentrations in values of µg/kg. Any analyte with a calculated concentration above the highest standard must be reanalyzed at a dilution that brings the concentration in the solution within the calibration curve. It is desirable to have the dilution fall within the top half of the calibration curve, but it is not required.

Calculations

A. Analyte Concentration

1. Low level

$$\text{Concentration } (\mu g/kg) \frac{(Ax)(Is)}{(Ais)(RRF)(Ws)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Amount of internal standard added in nanograms

Ws = Weight of sample purged

RRF = Relative response factor from the initial calibration

2. Medium/high level

$$Concentration\ (\mu g/kg) \frac{(Ax)\ (Is)\ (Vt)}{(Ais)\ (RRF)\ (Ws)\ (Vi)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Amount of internal standard added in nanograms

Vt = Volume of the total extract in microliters

Vi = Volume of the extract used for purging in microliters

Ws = Weight of sample extracted

RRF = Relative response factor from the initial calibration

B. QC Calculations:

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

C. Relative percent difference (RPD)

$$RPD = \frac{MSR - MSDR}{(1/2)\ (MSR + MSDR)} \times 100$$

Where:

MSR = Matrix spike measured concentration

MSDR = Matrix spike duplicate measured concentration

Statistical Information/Method Performance

The LCS must contain 80% to100% of the compounds in the calibration mix. LCS, MS, and surrogate recoveries and RPD are compared to the limits stored on the LIMS. These limits are statistically derived but must fall within 70% to 130% recovery for South Carolina compliance samples. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to 1-P-QM-QMA-9017309 for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

Each analysis batch must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD), or a laboratory control sample/laboratory control sample duplicate (LCS/LCSD). Additional QC samples may be required to meet project or state certification requirements. Every sample or QC analysis must contain internal standards and surrogate compounds.

The GC/MS system must be tuned to meet the criteria in Table 1 following BFB injection. The chromatographic conditions must be the same as those under which the samples are analyzed except that the rate of temperature ramping may be increased and the initial temperature and column flow may be different. The BFB tune must be verified every 12 hours.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards • Added to every sample, standard, method blank or QC sample	1. Peak areas within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If re-injecting meets the criteria, report this analysis. 3. If this reanalysis still shows the same problem, report results from first analysis and qualify data with a comment.

Quality Control Item	Acceptance Criteria	Corrective Action
Surrogates • Added to every sample, standard, method blank or QC sample	All % recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi-annual basis.	If non-compliant, check for calculation or preparation errors. If no errors are found, check system for problems and reanalyze. If this reanalysis still shows the same problem, report first analysis and qualify data with a comment. If recoveries are outside of specification high and no target compounds are detected then a reanalysis or comment is not required.
Method Blank (MB) Performed during each tune period after the initial calibration or CCV (minimum of 1 MB per 20 samples) • 1 scoop of Sodium Bisulfate is added if the preservative was added to the initial calibration.	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. Quantitative results for all target compounds must be less than the reporting limit for the associated samples.	1,2. Inspect system for possible problems and reanalyze. 3. If the MB contains target analytes and the associated samples do not, then no corrective action is required. If the target compounds in the MB are also in the associated samples, then they must be reanalyzed after a clean MB is obtained (certain projects may allow some exceptions for common laboratory contaminants like methylene chloride and acetone up to 5X the LOQ)
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) • LCS analyzed with each batch of ≤ 20 samples • LCSD analyzed if MS/MSD unavailable • Approximately 5 grams of sand is required for low level analysis. • 1 scoop of Sodium Bisulfate is added if the preservative was added to the initial calibration. • See Figures 2, 3 and 4 for preparation info.	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All % recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi-annual basis.	1,2. If non-compliant, check for calculation or preparation errors. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD re-analysis still fails, perform appropriate system maintenance and restart the tune period. Only with an LCS % recovery failing high (for the requested target compounds) with targets non-detected in the sample, can the results be reported. Otherwise, the sample must be analyzed with a compliant LCS.

Quality Control Item	Acceptance Criteria	Corrective Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD) • MS/MSD analyzed with each batch of ≤ 20 samples (if sufficient sample volume available) • See Figures 2 and 3 for preparation info.	1. % Recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi-annual basis 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries and/or RPDs outside of QC limits, consult the supervisor.

The method blank must meet all the above criteria for internal standard recoveries and surrogate recoveries. In addition, the method blank may not contain any target compound above the reporting limit for the associated samples. All method blanks must meet these criteria; otherwise, the system is considered out of control and corrective action must be taken.

If sufficient sample volume is not available to perform MS/MSD, LCS/LCSD are prepared and analyzed and must meet the above-mentioned criteria.

NOTE: Prior to release from the analytical laboratory, all data is reviewed in accordance with 1-P-QM-PRO-9015471.

The sample is analyzed using the same instrumental conditions as the standard (whether ICAL or CCV), tune and method blank. If the QA criteria are satisfied and no target compounds are detected at concentrations above the calibration range, the results can be reported. To avoid possible matrix effects, sample carryover and re-analyses, an initial dilution may be performed if:

1. Prescreening indicates a high volatile organic content in the sample

2. Historical data (or lack thereof) and/or sample appearance indicate a need for dilution

If target compounds are detected in the sample at concentrations above the calibration range, a dilution must be performed (see 1-P-QM-PRO-9015470 for information on when cleaning blanks must be run). See method SW-846 8260C for the recommended dilution procedures.

Attachment I

Gasoline Range Organics (GRO) by
Gas Chromatography/Mass Spectroscopy (GC/MS)

This section is specific to the steps required for GRO analysis. See the main body of the SOP for general information/ processes.

Basic Principles:

The GRO analysis is typically performed in conjunction with the analysis of other volatile target compounds by SW-846 Method 8260C. The GRO quantitation range is 0.1 minutes before the peak apex of C6 (hexane) to 0.2 minutes after the peak apex of C12 (dodecane); however, other ranges can be established. By establishing a (C12) GRO window to 0.2 minutes following the elution of dodecane, the areas from a trio of unresolved peaks eluting near to the upper limit of the range must consistently be included in the total GRO area. In addition, the range remains tight enough to ensure that no C13 or greater compounds can be included in the total GRO area. The C4 range retention time is determined by selecting the first peak after the air and/or artifact peak minus 0.1 minutes in the first level of standard analyzed with the ICAL. The C5 range retention time is 0.1 minutes before the peak apex of pentane.This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by GC/MS purge and trap methodologies. The area of the total ion chromatogram for the GRO range is determined. The area of the internal standards and surrogate standards are found and subtracted from the total area of the chromatogram within the desired time range. The resulting area is then quantitated versus the internal standard, fluorobenzene.

Interferences:

See main body of SOP.

Safety Precautions and Waste Handling:

See main body of SOP.

Personnel Training and Qualifications:

See main body of SOP.

NOTE: A separate Demonstration of Capability for GRO is required.

Sample Collection, Preservation, and Handling:

See main body of SOP.

Apparatus and Equipment:

See main body of SOP.

Reagents and Standards:

A. Reagents- See main body of SOP.

B. Standards- See main body of SOP for general standards.

1. GRO calibration standard – a 5500−µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier.

2. GRO QC standard – a 20,000−µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier

Store all standard solutions at −10° to −15°C

Calibration:

A. Initial calibration:

Prior to the analysis of any calibration level, retention time markers must be run for the GRO range of interest. The retention time markers are hexane (C6) and Dodecane (C12). Other markers can be used if different ranges are required by a project.

Internal standard calibration for GRO consists of analyzing six distinct levels of GRO area in order to produce a response factor for the GRO quantitation range of interest using the internal standard, fluorobenzene. The relative standard deviation of the response factor determines the suitability of the average relative response factor for calculation of the GRO concentration.

NOTE: 5 levels are required by the method.

1. Prepare the calibration standards at appropriate levels. Suggested calibration levels are 110, 220, 550, 1100, 2200, 4400 ppb. For low−level soils prepared according to EPA Method 5035, 1 scoop of sodium bisulfate is added to all calibration standards.

To prevent confusion and assure proper calibration, a Theoretical Standard Concentration (TSC) sheet is completed for each calibration (Figure 6). The TSC sheet contains the theoretical concentration for each certified analyte in the calibration at the various levels.

2. Each level is analyzed as described in the procedure under data analysis. Next, tabulate the area response for the GRO quantitation range minus the peak areas for the internal and surrogate standards that elute within the GRO range. Calculate the relative response factor (RRF) for GRO (see Calculation section) using the internal standard peak area for fluorobenzene.

NOTE: Although four internal standard compounds are spiked for the 8260B analysis, only Fluorobenzene, is used for the quantitation of the GRO result.

3. Calculate the average relative response factors for the GRO quantitation range of interest. The calibration levels are evaluated on the basis of the relative standard deviation of the RRF values (% RSD). The %RSD for the GRO range of interest must be ≤20%. If the calibration meets this requirement, then the average RRF is used to calculate sample concentrations. If the %RSD is >20% then re-analysis of one or more levels can be necessary before the calibration is valid.

B. Initial Calibration Verification (ICV):

Following the calibration, an Initial Calibration Verification (ICV) standard must be run. The ICV is prepared according to the TSC sheet in Figure 6 (QC prep). The ICV acts as a second source standard to check against the initial calibration. Results must quantitate within the 70-130% window. If the ICV does not meet the aforementioned criteria, a second ICV can be run before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

C. Continuing calibration verification (CCV):

The CCV involves an analysis for the 1100-ppb standard. The calibration is considered valid if the percent drift is ≤20%. Also, the internal standard peak area of fluorobenzene for the CCV is monitored against the mid-point standard of the initial calibration and must be –50% to +100% of the area counts. If any criteria listed above fails, the CCV is considered invalid. In the case where two consecutive CCVs fail, corrective action must be taken which can include re-analysis of the calibration check, instrument maintenance, and/or recalibration. If the criteria are met, the selected quantitation method from the initial calibration is used for blank and sample calculations until the end of the 12-hour period.

Procedure:

Samples must be analyzed in accordance with the analyses listed in the main body of this SOP. However, additional requirements are required for the GRO data analysis.

A. The Total Ion Chromatogram (TIC) is reviewed to insure proper integration around the 8260 surrogates and internal standards. Also the TIC is checked to make sure all major peaks are integrated.

B. The quantitation of the GRO range is performed using the equations listed in the Calculations section of this procedure. All calculations must report concentrations in values of µg/L. In the case where the total GRO concentration exceeds the calibration range, the sample is re-analyzed at a dilution that brings the GRO concentration within the calibration range of the GC/MS system.

Calculations:

See main body of SOP.

Statistical Information/Method Performance:

See main body of SOP.

Quality Assurance/Quality Control:

See main body of SOP.

Table 1

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	base peak, 100% relative abundance
96	5% to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5% to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table 2

Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
1,1-Dichloroethene	96	61, 63

Compound Name	Primary Ion	Secondary Ion
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
trans-1,2-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77, 110
trans-1,3-Dichloropropene	75	77, 110
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzen-d4	152	115

Continued
Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66

Compound Name	Primary Ion	Secondary Ion
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
trans-1,2-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77, 110
trans-1,3-Dichloropropene	75	77, 110
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzene-d4	152	115

Table 3
Minimum Relative Response Factors For ICAL and CCV

Volatile Compounds	Minimum Response Factor
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100

Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon Disulfide	0.100
Methyl Acetate	0.100
Methylene Chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100

Table 3 Continued

Volatile Compounds	Minimum Response Factor
m&p-Xylene	0.100
o-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Attachment:

End of document

Version history

Version	Approval	Revision information
4	24.JUN.2016	

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
EPA SW846 Method 8260B/C

Date:_____
Instrument:_____

VOA1= 1:5 dilution of VCS#1B, VCS#2B, and VCS#4C
VOA2= 1:5 dilution of VCS#2B
VOA3= 1:5 dilution of VCS#3B and Vacrolein
VOA6= 1:5 dilution of VCS#6
2CEVE= 1:5 dilution of VCS#1B-2CEVE
VOA8= 1:5 dilution of Hexachloroethane and 2,2'-oxybis(1-Chloropropane)

Stock mix name	VOA1 _____ 2CEVE _____ 1,3-BUT _____	VOA3 _____	VOA2 _____	VOA6 _____ EE _____ Custom V LG Freon _____	n-PEN _____ VOA8 _____	CYC _____	EOH _____	Restek Gases (2000 ppm) Lt#_____	IH826SS 250 ppm @ _____ 2500 ppm 8260SS $ ----------	Flask mL	MeOH mL Lt#____
300 ppb std	15 µL	6 µL		15µL	15 µL	30 µL	60 µL	7.5 µL	60µL@/6µL$	50	1
100 ppb std	5 µL	2 µL		5 µL	5 µL	10 µL	20 µL	2.5 µL	20µL/2µL$	50	1
50 ppb std	5 µL	2 µL		5 µL	5 µL	10 µL	20 µL	2.5 µL	20µL @/ 2µL$	100	2
20 ppb std	4 µL	1.6 µL	4 µL	4 µL	4 µL	16 µL	32 µL	2.0 µL	16µL@/1.6µL$	200	4
10 ppb std	2 µL	0.8 µL	2 µL	2 µL	2 µL	8 µL	16 µL	1.0 µL	8µL@/ 0.8µL$	200	4
4 ppb std	4 µL	1.6 µL	12 µL	4 µL	4 µL	32 µL	40 µL	2.0 µL	16µL@/ 1.6µL$	1000 *	20
1 ppb std	* Aliquot 12.5 mL of 1000 mL flask into 50 mL flask										
0.5 ppb MDL std	+ Aliquot 12.5 mL of 1000 mL flask into 100 mL flask										

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb	0.5 ppb
Benzene	CS#1B	5000	300	100	50	20	10	4	1	0.5
Bromobenzene		5000	300	100	50	20	10	4	1	0.5
Bromodichloromethane		5000	300	100	50	20	10	4	1	0.5
Bromoform		5000	300	100	50	20	10	4	1	0.5
n-Butylbenzene		5000	300	100	50	20	10	4	1	0.5
sec-Butylbenzene		5000	300	100	50	20	10	4	1	0.5
tert-Butylbenzene		5000	300	100	50	20	10	4	1	0.5
Carbon Tetrachloride		5000	300	100	50	20	10	4	1	0.5
Chlorobenzene		5000	300	100	50	20	10	4	1	0.5
Chloroform		5000	300	100	50	20	10	4	1	0.5
2-Chlorotoluene		5000	300	100	50	20	10	4	1	0.5
4-Chlorotoluene		5000	300	100	50	20	10	4	1	0.5
Dibromochloromethane		5000	300	100	50	20	10	4	1	0.5
1,2-Dibromo-3-chloropropane		5000	300	100	50	20	10	4	1	0.5
1,2-Dibromoethane (EDB)		5000	300	100	50	20	10	4	1	0.5
Dibromomethane		5000	300	100	50	20	10	4	1	0.5
1,2-Dichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,3-Dichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,4-Dichlorobenzene		5000	300	100	50	20	10	4	1	0.5

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
EPA SW846 Method 8260A/B

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb	0.5 ppb
1,1-Dichloroethane	CS#1B	5000	300	100	50	20	10	4	1	0.5
1,2-Dichloroethane		5000	300	100	50	20	10	4	1	0.5
1,1-Dichloroethene		5000	300	100	50	20	10	4	1	0.5
cis-1,2-Dichloroethene		5000	300	100	50	20	10	4	1	0.5
trans-1,2-Dichloroethene		5000	300	100	50	20	10	4	1	0.5
1,2-Dichloropropane		5000	300	100	50	20	10	4	1	0.5
1,3-Dichloropropane		5000	300	100	50	20	10	4	1	0.5
2,2-Dichloropropane		5000	300	100	50	20	10	4	1	0.5
1,1-Dichloropropene		5000	300	100	50	20	10	4	1	0.5
cis-1,3-Dichloropropene		5000	300	100	50	20	10	4	1	0.5
trans-1,3-Dichloropropene		5000	300	100	50	20	10	4	1	0.5
Ethylbenzene		5000	300	100	50	20	10	4	1	0.5
Hexachlorobutadiene		5000	300	100	50	20	10	4	1	0.5
Isopropylbenzene		5000	300	100	50	20	10	4	1	0.5
p-Isopropyltoluene		5000	300	100	50	20	10	4	1	0.5
Methylene Chloride		5000	300	100	50	20	10	4	1	0.5
Naphthalene		5000	300	100	50	20	10	4	1	0.5
n-Propylbenzene		5000	300	100	50	20	10	4	1	0.5
Styrene		5000	300	100	50	20	10	4	1	0.5
1,1,1,2-Tetrachloroethane		5000	300	100	50	20	10	4	1	0.5
1,1,2,2-Tetrachloroethane		5000	300	100	50	20	10	4	1	0.5
Tetrachloroethene		5000	300	100	50	20	10	4	1	0.5
Toluene		5000	300	100	50	20	10	4	1	0.5
1,2,3-Trichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,2,4-Trichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,3,5-Trichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,1,1-Trichloroethane		5000	300	100	50	20	10	4	1	0.5
1,1,2-Trichloroethane		5000	300	100	50	20	10	4	1	0.5
Trichloroethene		5000	300	100	50	20	10	4	1	0.5
1,2,3-Trichloropropane		5000	300	100	50	20	10	4	1	0.5
1,2,4-Trimethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,3,5-Trimethylbenzene		5000	300	100	50	20	10	4	1	0.5
m-Xylene		5000	300	100	50	20	10	4	1	0.5
o-Xylene		5000	300	100	50	20	10	4	1	0.5
p-Xylene		5000	300	100	50	20	10	4	1	0.5
1-Chlorohexane		5000	300	100	50	20	10	4	1	0.5
Pentachloroethane	CS#6	5000	300	100	50	20	10	4	1	0.5
Allyl Chloride		5000	300	100	50	20	10	4	1	0.5
Bromochloromethane		5000	300	100	50	20	10	4	1	0.5
Methyl Acetate		5000	300	100	50	20	10	4	1	0.5
Methylcyclohexane		5000	300	100	50	20	10	4	1	0.5
2-Methylnaphthalene		5000	300	100	50	20	10	4	1	0.5
1,2,3-Trimethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,2-Diethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,3-Diethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,4-Diethylbenzene		5000	300	100	50	20	10	4	1	0.5

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
EPA SW846 Method 8260A/B

Compound name	Std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb	0.5 ppb
Methacrylonitrile	CS#2B	12500	750	250	125	100	50	40	10	5
Propionitrile		25000	1500	500	250	200	100	80	20	10
trans-1,4-Dichloro-2-Butene		12500	750	250	125	100	50	40	10	5
t-Butyl Alcohol		25000	1500	500	250	200	100	80	20	10
2-Propanol		25000	1500	500	250	200	100	80	20	10
Isobutyl Alcohol		62500	3750	1250	625	500	250	200	50	25
n-Butanol		125000	7500	2500	1250	1000	500	400	100	50
1,4-Dioxane		62500	3750	1250	625	500	250	200	50	25
2-Butanone	CS#3B	25000	600	200	100	40	20	8	2	1
2-Hexanone		25000	600	200	100	40	20	8	2	1
4-Methyl-2-Pentanone		25000	600	200	100	40	20	8	2	1
Acetone		25000	600	200	100	40	20	8	2	1
Acrylonitrile		12500	300	100	50	20	10	4	1	0.5
2-Nitropropane		25000	600	200	100	40	20	8	2	1
Tetrahydrofuran		25000	600	200	100	40	20	8	2	1
Methyl-t-butyl Ether	CS#4C	5000	300	100	50	20	10	4	1	0.5
Ethyl Methacrylate		5000	300	100	50	20	10	4	1	0.5
Methyl Methacrylate		5000	300	100	50	20	10	4	1	0.5
Freon 113		5000	300	100	50	20	10	4	1	0.5
Hexane		5000	300	100	50	20	10	4	1	0.5
Heptane		5000	300	100	50	20	10	4	1	0.5
Cyclohexane		5000	300	100	50	20	10	4	1	0.5
Benzyl Chloride		5000	300	100	50	20	10	4	1	0.5
Methyl Iodide		5000	300	100	50	20	10	4	1	0.5
Carbon Disulfide		5000	300	100	50	20	10	4	1	0.5
2-Chloro-1,3-Butadiene		5000	300	100	50	20	10	4	1	0.5
di-Isopropyl Ether		5000	300	100	50	20	10	4	1	0.5
tert-Amyl Methyl Ether		5000	300	100	50	20	10	4	1	0.5
Ethyl-t-butyl Ether		5000	300	100	50	20	10	4	1	0.5
Bromomethane	Gas	2000	300	100	50	20	10	4	1	0.5
Chloroethane	mix	2000	300	100	50	20	10	4	1	0.5
Chloromethane		2000	300	100	50	20	10	4	1	0.5
Dichlorodifluoromethane		2000	300	100	50	20	10	4	1	0.5
Trichlorofluoromethane		2000	300	100	50	20	10	4	1	0.5
Vinyl Chloride		2000	300	100	50	20	10	4	1	0.5
Cyclohexanone	CYC	6250	3750	1250	625	500	250	200	50	25
2-Chloroethyl Vinyl Ether	2CEVE	5000	300	100	50	20	10	4	1	0.5
1,3-Butadiene	1,3-BUT	1000	300	100	50	20	10	4	1	0.5

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260C

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb
Ethyl Ether	EE	1000	300	100	50	20	10	4	1
n-Pentane	n-PEN	1000	300	100	50	20	10	4	1
Acrolein	VACR	125000	3000	1000	500	200	100	40	10
Cyclohexanone	CYC	6250	3750	1250	625	500	250	200	50
Ethanol	EOH	12500	15000	5000	2500	2000	1000	500	125
Dichlorofluoromethane	Custom	1000	300	100	50	20	10	4	1
Freon 123a	V Freon	1000	300	100	50	20	10	4	1

ppb of analytical standard = (stock ppm)(µL stock) / flask mL

Analyst:_____
Date:_____

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

QVOA1= 1:25 QCS#1B, QCS#2B, QCS3B,QCS#4C Date:_____
QVOA6 = 1: 25 QCS#6 QGASES=1:50 Restek 502.2 "Q" Gas mix Instrument:_____
QARC = 1: 25 QCS#1B2CEVE, QACR stock QVOA8= 1:25 VOA8
Qn-pentane = 40ul of n-pentane lot#_____ to 960ul MEOH lot# _____
QBUT = 40ul of 1,3-Butadiene lot#_____ to 960ul MEOH lot# _____
Custom Freon Q = 40ul of Custom Q LG Freon lot#_____ to 960 ul MEOH lot#_____

Stock mix Name	QVOA1 _____ QARC _____ QBUT _____	QVOA6 _____ QEE _____	QCYC _____	QEOH _____	8260 SS 2500 ppm Lot#_____ MeOH Prep Only	QGASES _____ Qn-pentane QVOA8 _____ _____ Custom Q LG Freon _____	Final Volume	MeOH Lot# _____ MeOH Prep Only	Used
20 ppb	2.5 µL	2.5 µL	2.5 µL	5.0 µL	0.1 ul	2.5 µL	5 mL Syringe	.1 mL	
20 ppb	21.5 µL	21.5 µL	21.5 µL	43.0 µL	-	21.5 µL	43 mL Vial	-	
20 ppb	25.0 µL	25.0 µL	25.0 µL	50.0 µL	1.0 ul	25.0 µL	50 mL Flask	1 mL	

Compound name	std mix	Stock ppm	20 ppb
Benzene	QCS#1B	1000	20
Bromobenzene		1000	20
Bromodichloromethane		1000	20
Bromoform		1000	20
n-Butylbenzene		1000	20
sec-Butylbenzene		1000	20
tert-Butylbenzene		1000	20
Carbon Tetrachloride		1000	20
Chlorobenzene		1000	20
Chloroform		1000	20
2-Chlorotoluene		1000	20
4-Chlorotoluene		1000	20
Dibromochloromethane		1000	20
1,2-Dibromo-3-chloropropane		1000	20
1,2-Dibromoethane (EDB)		1000	20
Dibromomethane		1000	20
1,2-Dichlorobenzene		1000	20
1,3-Dichlorobenzene		1000	20
1,4-Dichlorobenzene		1000	20
1,1-Dichloroethane		1000	20
1,2-Dichloroethane		1000	20
1,1-Dichloroethene		1000	20
cis-1,2-Dichloroethene		1000	20
trans-1,2-Dichloroethene		1000	20
1,2-Dichloropropane		1000	20
1,3-Dichloropropane		1000	20
2,2-Dichloropropane		1000	20
1,1-Dichloropropene		1000	20
cis-1,3-Dichloropropene		1000	20

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
trans-1,3-Dichloropropene	QCS#1B	1000	20
Ethylbenzene		1000	20
Hexachlorobutadiene		1000	20
p-Isopropyltoluene		1000	20
Methylene Chloride		1000	20
Isopropylbenzene (Cumene)		1000	20
Naphthalene		1000	20
n-Propylbenzene		1000	20
Styrene		1000	20
1,1,1,2-Tetrachloroethane		1000	20
1,1,2,2-Tetrachloroethane		1000	20
Tetrachloroethene		1000	20
Toluene		1000	20
1,2,3-Trichlorobenzene		1000	20
1,2,4-Trichlorobenzene		1000	20
1,3,5-Trichlorobenzene		1000	20
1,1,1-Trichloroethane		1000	20
1,1,2-Trichloroethane		1000	20
Trichloroethene		1000	20
1,2,3-Trichloropropane		1000	20
1,2,4-Trimethylbenzene		1000	20
1,3,5-Trimethylbenzene		1000	20
m-Xylene		1000	20
o-Xylene		1000	20
p-Xylene		1000	20
1-Chlorohexane		1000	20
Bromomethane	QGas	2000	20
Chloroethane	mix	2000	20
Chloromethane		2000	20
Dichlorodifluoromethane		2000	20
Trichlorofluoromethane		2000	20
Vinyl Chloride		2000	20
Methacrylonitrile	QCS#2B	7500	150
Propionitrile		7500	150
trans-1,4-Dichloro-2-Butene		5000	100
t-Butyl Alcohol		10000	200
2-Propanol		7500	150
Isobutyl Alcohol		25000	500
n-Butanol		50000	1000
1,4-Dioxane		25000	500
2-Butanone	QCS#3B	7500	150
2-Hexanone		5000	100

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
4-Methyl-2-Pentanone	QCS#3B	5000	100
Acetone		7500	150
Acrylonitrile		5000	100
2-Nitropropane		1000	20
Tetrahydrofuran		5000	100
Methyl-t-butyl Ether	QCS#4C	1000	20
Ethyl Methacrylate		1000	20
Methyl Methacrylate		1000	20
Freon 113		1000	20
Hexane		1000	20
Heptane		1000	20
Cyclohexane		1000	20
Benzyl Chloride		1000	20
Methyl Iodide		1000	20
Carbon Disulfide		1000	20
2-Chloro-1,3-Butadiene		1000	20
di-Isopropyl Ether		1000	20
tert-Amyl Methyl Ether		1000	20
Ethyl-t-butyl Ether		1000	20
Pentachloroethane	QCS#6	1000	20
Allyl Chloride		1000	20
Bromochloromethane		1000	20
Methyl Acetate		1000	20
Methylcyclohexane		1000	20
2-Methylnaphthalene		1000	20
1,2,3-Trimethylbenzene		1000	20
1,2-Diethylbenzene		1000	20
1,3-Diethylbenzene		1000	20
1,4-Diethylbenzene		1000	20
Acrolein	QACR	7500	150
2- Chloroethyl Vinyl Ether	QCS#1B 2CEVE	1000	20
Cyclohexanone	QCYC	1000	500
Ethyl Ether	QEE	40	20
n-Pentane	Qn-PEN	40	20
1,3-Butadiene	QBUT	40	20

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
Dichlrofluoromethane	Custom Q	1000	20
Freon 123a	LG Freon	1000	20
Ethanol	QEOH	1000	1000
Hexachloroethane	QVOA8	1000	20
2,2'-oxybis(1-chloropropane)		1000	20

ppb of analytical standard = (stock ppm)(µl stock) / final volume

Analyst:_____
Date:_____

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
High Level Prep with QC

Prep date:_____
Instrument:_____
exp. date:_____

*LCS/LCSD = DF 50 of final volume

Stock mix name	QCS#1B Lt#_____ QCS#3B Lt#_____ QCS#4C Lt#_____ QCS#1B-2CEVE Lt#_____ QCS#2B Lt#_____ QCS#6 Lt#_____	QACR _____ EE _____ Custom Q LG Freon Lot #____	CYC _____	EtOH _____	2000 ppm Restek 502.2 "Q" Gas mix Lt#_____	8260 SS $ Lt#_____ 2500 ppm $	Final Volume * MeOH Lot# _____
High Level	10 µL	10 µL	40 µL	40 µL	5 µL	10 µL	10 mL

Compound name	std mix	Stock ppm	20 ppb
Benzene	QCS#1B	1000	20
Bromobenzene		1000	20
Bromodichloromethane		1000	20
Bromoform		1000	20
n-Butylbenzene		1000	20
sec-Butylbenzene		1000	20
tert-Butylbenzene		1000	20
Carbon Tetrachloride		1000	20
Chlorobenzene		1000	20
Chloroform		1000	20
2-Chlorotoluene		1000	20
4-Chlorotoluene		1000	20
Dibromochloromethane		1000	20
1,2-Dibromo-3-chloropropane		1000	20
1,2-Dibromoethane (EDB)		1000	20
Dibromomethane		1000	20
1,2-Dichlorobenzene		1000	20
1,3-Dichlorobenzene		1000	20
1,4-Dichlorobenzene		1000	20
1,1-Dichloroethane		1000	20
1,2-Dichloroethane		1000	20
1,1-Dichloroethene		1000	20
cis-1,2-Dichloroethene		1000	20
trans-1,2-Dichloroethene		1000	20
1,2-Dichloropropane		1000	20
1,3-Dichloropropane		1000	20
2,2-Dichloropropane		1000	20
1,1-Dichloropropene		1000	20
cis-1,3-Dichloropropene		1000	20
trans-1,3-Dichloropropene		1000	20
Ethylbenzene		1000	20

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
High Level Prep with QC

Compound name	std mix	Stock ppm	20 ppb
Hexachlorobutadiene	QCS#1B	1000	20
Isopropylbenzene (Cumene)		1000	20
p-Isopropyltoluene		1000	20
Methylene Chloride		1000	20
Naphthalene		1000	20
n-Propylbenzene		1000	20
Styrene		1000	20
1,1,1,2-Tetrachloroethane		1000	20
1,1,2,2-Tetrachloroethane		1000	20
Tetrachloroethene		1000	20
Toluene		1000	20
1,2,3-Trichlorobenzene		1000	20
1,2,4-Trichlorobenzene		1000	20
1,3,5-Trichlorobenzene		1000	20
1,1,1-Trichloroethane		1000	20
1,1,2-Tricholroethane		1000	20
Trichloroethene		1000	20
1,2,3-Trichloropropane		1000	20
1,2,4-Trimethylbenzene		1000	20
1,3,5-Trimethylbenzene		1000	20
m-Xylene		1000	20
o-Xylene		1000	20
p-Xylene		1000	20
1-Chlorohexane		1000	20
2-Chloroethyl Vinyl Ether	QCS#1B 2CLEVE	1000	20
Bromomethane	Gas	2000	20
Chloroethane	mix	2000	20
Chloromethane		2000	20
Dichlorodifluoromethane		2000	20
Trichlorofluoromethane		2000	20
Vinyl Chloride		2000	20
Methacrylonitrile	QCS#2B	7500	150
Propionitrile		7500	150
trans-1,4-Dichloro-2-Butene		5000	100
t-Butyl Alcohol		10000	200
2-Propanol		7500	150
Isobutyl Alcohol		25000	500
n-Butanol		50000	1000
1,4-Dioxane		25000	500

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
High Level Prep with QC

Compound name	std mix	Stock ppm	20 ppb
2-Hexanone	QCS#3B	5000	100
2-Butanone		7500	150
4-Methyl-2-Pentanone		5000	100
Acetone		7500	150
Acrylonitrile		5000	100
2-Nitropropane		1000	20
Tetrahydrofuran		5000	100
Methyl-t-butyl Ether	QCS#4C	1000	20
Ethyl Methacrylate		1000	20
Methyl Methacrylate		1000	20
Freon 113		1000	20
Hexane		1000	20
Heptane		1000	20
Cyclohexane		1000	20
Benzyl Chloride		1000	20
Methyl Iodide		1000	20
Carbon Disulfide		1000	20
2-Chloro-1,3-Butadiene		1000	20
di-Isopropyl Ether		1000	20
tert-Amyl Methyl Ether		1000	20
Ethyl-t-butyl Ether		1000	20
Pentachloroethane	QCS#6	1000	20
Allyl Chloride		1000	20
Bromochloromethane		1000	20
2-Methylnaphthalene		1000	20
Methyl Acetate		1000	20
Methylcyclohexane		1000	20
1,2,3-Trimethylbenzene		1000	20
1,2-Diethylbenzene		1000	20
1,3-Diethylbenzene		1000	20
1,4-Diethylbenzene		1000	20

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
High Level Prep with QC

Compound name	std mix	Stock ppm	20 ppb
Freon 123a	Custom Q	1000	20
Dichlorofluoromethane	LG Freon	1000	20
Acrolein	QACR	7500	150
Cyclohexanone	CYC	6250	500
Ethanol	EtOH	12500	1000
Ethyl Ether	EE	1000	20

ppb of analytical standard = (stock ppm)(µl stock) / final volume

Analyst:_____
Date:_____

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
MeOH Prep

Prepped:_____
Instrument:_____
exp. date:_____

QVOA6= 1:25 QCS#6
QARC = 1: 25 QCS#1B2CEVE, QACR stock
QVOA1= 1:25 QCS#1B, QCS#2B, QCS3B, QCS#4C QGASES=1:50 Restek 502.2 "Q" Gas mix
QCustom LG Freon = 40uL of Custom Q LG Freon lot#_____to 960uL MEOH lot#_____
Qn-pentane = 40ul of n-pentane lot#_____ to 960ul MEOH lot# _____

Stock mix name	QVOA1 _____ QARC _____ QCUSTOM LG FREON _____	QVOA6 _____ QEE _____ QVOA8 _____	QGASES _____ QCYC _____ Q n-Pentane _____	QEOH _____	826SS $ _____ 25 ppm $ 8260 SS@ _____ 2500ppm @	Final Volume MeOH Lot#_____	Prep Used
As Rec'd Med	250 µL	250 µL	250 ul	500 µl	1.0 mL $	10 mL MeOH	
High Level **NO QC**	250 µL	250 µL	250 ul	500 µL	10 µL @	10 mL MeOH	

Compound name	std mix	Stock ppm	20 ppb
Benzene	QCS#1B	1000	20
Bromobenzene		1000	20
Bromodichloromethane		1000	20
Bromoform		1000	20
n-Butylbenzene		1000	20
sec-Butylbenzene		1000	20
tert-Butylbenzene		1000	20
Carbon Tetrachloride		1000	20
Chlorobenzene		1000	20
Chloroform		1000	20
2-Chlorotoluene		1000	20
4-Chlorotoluene		1000	20
Dibromochloromethane		1000	20
1,2-Dibromo-3-chloropropane		1000	20
1,2-Dibromoethane (EDB)		1000	20
Dibromomethane		1000	20
1,2-Dichlorobenzene		1000	20
1,3-Dichlorobenzene		1000	20
1,4-Dichlorobenzene		1000	20
1,1-Dichloroethane		1000	20
1,2-Dichloroethane		1000	20
1,1-Dichloroethene		1000	20
cis-1,2-Dichloroethene		1000	20
trans-1,2-Dichloroethene		1000	20
1,2-Dichloropropane		1000	20
1,3-Dichloropropane		1000	20
2,2-Dichloropropane		1000	20
1,1-Dichloropropene		1000	20
cis-1,3-Dichloropropene		1000	20

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
trans-1,3-Dichloropropene	QCS#1B	1000	20
Ethylbenzene		1000	20
Hexachlorobutadiene		1000	20
Isopropylbenzene (Cumene)		1000	20
p-Isopropyltoluene		1000	20
Methylene Chloride		1000	20
Naphthalene		1000	20
n-Propylbenzene		1000	20
Styrene		1000	20
1,1,1,2-Tetrachloroethane		1000	20
1,1,2,2-Tetrachloroethane		1000	20
Tetrachloroethene		1000	20
Toluene		1000	20
1,2,3-Trichlorobenzene		1000	20
1,2,4-Trichlorobenzene		1000	20
1,3,5-Trichlorobenzene		1000	20
1,1,1-Trichloroethane		1000	20
1,1,2-Trichloroethane		1000	20
Trichloroethene		1000	20
1,2,3-Trichloropropane		1000	20
1,2,4-Trimethylbenzene		1000	20
1,3,5-Trimethylbenzene		1000	20
m-Xylene		1000	20
o-Xylene		1000	20
p-Xylene		1000	20
1-Chlorohexane		1000	20
Bromomethane	Gas	2000	20
Chloroethane	mix	2000	20
Chloromethane		2000	20
Dichlorodifluoromethane		2000	20
Trichlorofluoromethane		2000	20
Vinyl Chloride		2000	20
Methacrylonitrile	QCS#2B	7500	150
Propionitrile		7500	150
trans-1,4-Dichloro-2-Butene		5000	100
t-Butyl Alcohol		10000	200
2-Propanol		7500	150
Isobutyl Alcohol		25000	500
n-Butanol		50000	1000
1,4-Dioxane		25000	500
2-Hexanone	QCS#3B	5000	100
4-Methyl-2-Pentanone		5000	100

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
Acetone	QCS#3B	7500	150
Acrylonitrile		5000	100
2-Nitropropane		1000	20
Tetrahydrofuran		5000	100
2-Butanone		7500	150
Methyl-t-butyl Ether	QCS#4C	1000	20
Ethyl Methacrylate		1000	20
Methyl Methacrylate		1000	20
Freon 113		1000	20
Hexane		1000	20
Heptane		1000	20
Cyclohexane		1000	20
Benzyl Chloride		1000	20
Methyl Iodide		1000	20
Carbon Disulfide		1000	20
2-Chloro-1,3-Butadiene		1000	20
di-Isopropyl Ether		1000	20
tert-Amyl Methyl Ether		1000	20
Ethyl-t-butyl Ether		1000	20
2-Chloroethyl Vinyl Ether	QCS#1B 2CEVE	1000	20
Pentachloroethane	QCS#6	1000	20
Allyl Chloride		1000	20
Bromochloromethane		1000	20
2-Methylnaphthalene		1000	20
Methyl Acetate		1000	20
Methylcyclohexane		1000	20
1,2,3-Trimethylbenzene		1000	20
1,2-Diethylbenzene		1000	20
1,3-Diethylbenzene		1000	20
1,4-Diethylbenzene		1000	20
Ethanol	QEOH	1000	1000
Cyclohexanone	QCYC	1000	500
Ethyl Ether	QEE	40	20
Hexachloroethane	QVOA8	1000	20
2,2'-oxybis(1-chloropropane)		1000	20

Theoretical Standard Concentrations
Quality Control for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
Dichlorofluoromethane	QCUSTOM	1000	20
Freon 133a	LG FREON	1000	20
Acrolein	QACR	7500	150
n-Pentane	Q n-Penane	40	20

ppb of analytical standard = (stock ppm)(µl stock) / final volume .

Analyst:_____
Date:_____

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

Date:_____
Instrument:_____

Qn-pentane = 40ul of n-pentane lot#_____ to 960ul MEOH lot# _____
QBUT = 40ul of 1,3-Butadiene lot#_____ to 960ul MEOH lot# _____

Stock mix Name	QCS#1B _____ QCS#2B _____ QCS#3B _____ QCS#4C _____ QACR _____	QCYC _____ QEE _____ QBUT _____ Qn-pentane _____	QEOH _____	8260 SS 2500 ppm Lot#_____ QCS#1B 2CEVE _____ QCS#6 _____ Cust. Q LG Freon _____	Restek 502.2 "Q" Gas mix _____	Final Volume	MeOH Lot# _____	Used
20 ppb	2.0 µL	50.0 µL	100.0 µL	2.0 ul	1.0 µL	100 mL Flask	2 mL	

Compound name	std mix	Stock ppm	20 ppb
Benzene	QCS#1B	1000	20
Bromobenzene		1000	20
Bromodichloromethane		1000	20
Bromoform		1000	20
n-Butylbenzene		1000	20
sec-Butylbenzene		1000	20
tert-Butylbenzene		1000	20
Carbon Tetrachloride		1000	20
Chlorobenzene		1000	20
Chloroform		1000	20
2-Chlorotoluene		1000	20
4-Chlorotoluene		1000	20
Dibromochloromethane		1000	20
1,2-Dibromo-3-chloropropane		1000	20
1,2-Dibromoethane (EDB)		1000	20
Dibromomethane		1000	20
1,2-Dichlorobenzene		1000	20
1,3-Dichlorobenzene		1000	20
1,4-Dichlorobenzene		1000	20
1,1-Dichloroethane		1000	20
1,2-Dichloroethane		1000	20
1,1-Dichloroethene		1000	20
cis-1,2-Dichloroethene		1000	20
trans-1,2-Dichloroethene		1000	20
1,2-Dichloropropane		1000	20
1,3-Dichloropropane		1000	20
2,2-Dichloropropane		1000	20
1,1-Dichloropropene		1000	20

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
cis-1,3-Dichloropropene	QCS#1B	1000	20
trans-1,3-Dichloropropene		1000	20
Ethylbenzene		1000	20
Hexachlorobutadiene		1000	20
p-Isopropyltoluene		1000	20
Methylene Chloride		1000	20
Isopropylbenzene (Cumene)		1000	20
Naphthalene		1000	20
n-Propylbenzene		1000	20
Styrene		1000	20
1,1,1,2-Tetrachloroethane		1000	20
1,1,2,2-Tetrachloroethane		1000	20
Tetrachloroethene		1000	20
Toluene		1000	20
1,2,3-Trichlorobenzene		1000	20
1,2,4-Trichlorobenzene		1000	20
1,3,5-Trichlorobenzene		1000	20
1,1,1-Trichloroethane		1000	20
1,1,2-Trichloroethane		1000	20
Trichloroethene		1000	20
1,2,3-Trichloropropane		1000	20
1,2,4-Trimethylbenzene		1000	20
1,3,5-Trimethylbenzene		1000	20
m-Xylene		1000	20
o-Xylene		1000	20
p-Xylene		1000	20
1-Chlorohexane		1000	20
Bromomethane	QGas	2000	20
Chloroethane	mix	2000	20
Chloromethane		2000	20
Dichlorodifluoromethane		2000	20
Trichlorofluoromethane		2000	20
Vinyl Chloride		2000	20
Methacrylonitrile	QCS#2B	7500	150
Propionitrile		7500	150
trans-1,4-Dichloro-2-Butene		5000	100
t-Butyl Alcohol		10000	200
2-Propanol		7500	150
Isobutyl Alcohol		25000	500
n-Butanol		50000	1000
1,4-Dioxane		25000	500
2-Butanone	QCS#3B	7500	150

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
2-Hexanone	QCS#3B	5000	100
4-Methyl-2-Pentanone		5000	100
Acetone		7500	150
Acrylonitrile		5000	100
2-Nitropropane		1000	20
Tetrahydrofuran		5000	100
Methyl-t-butyl Ether	QCS#4C	1000	20
Ethyl Methacrylate		1000	20
Methyl Methacrylate		1000	20
Freon 113		1000	20
Hexane		1000	20
Heptane		1000	20
Cyclohexane		1000	20
Benzyl Chloride		1000	20
Methyl Iodide		1000	20
Carbon Disulfide		1000	20
2-Chloro-1,3-Butadiene		1000	20
di-Isopropyl Ether		1000	20
tert-Amyl Methyl Ether		1000	20
Ethyl-t-butyl Ether		1000	20
Pentachloroethane	QCS#6	1000	20
Allyl Chloride		1000	20
Bromochloromethane		1000	20
Methyl Acetate		1000	20
Methylcyclohexane		1000	20
2-Methylnaphthalene		1000	20
1,2,3-Trimethylbenzene		1000	20
1,2-Diethylbenzene		1000	20
1,3-Diethylbenzene		1000	20
1,4-Diethylbenzene		1000	20
Acrolein	QACR	7500	150
2- Chloroethyl Vinyl Ether	QCS#1B 2CEVE	1000	20
Cyclohexanone	QCYC	1000	500
Ethyl Ether	QEE	40	20
n-Pentane	Qn-PEN	40	20
1,3-Butadiene	QBUT	40	20

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B/C
Low Soil and NJ MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
Ethanol	QEOH	1000	1000
Dichlorofluoromethane	Custom Q	1000	20
Freon 123a	LG Freon	1000	20

ppb of analytical standard = (stock ppm)(µl stock) / final volume

Analyst:_____
Date:_____

Theoretical Standard Concentrations
Gasoline Range Organics
Low Level / High Level Soil Prep

Instrument:_____
Restek V Unleaded Gasoline Composite Standard Date:_____

Lot: _____ Exp: _____
8260 SS Lt#_____

INITIAL CALIBRATION TSC			level 6	level 5	level 4	level 3	level 2	level 1	MDL
		ul stock	40	20	10	10	4	2	4
		ul SS stock	1	1	1	2	2	2	10
		FV H20 Ml	50	50	50	100	100	100	500
Compound Name	**CAS #**	**Stock ppm**	**Conc. ug/Kg**	**Conc. ug/Kg**	**Conc. ug/Kg**	**Conc. ug/Kg**	**Conc. ug/Kg**	**Conc. ug/Kg**	**Conc. ug/Kg**
Unleaded Gasoline Composite Std.	8006-61-9	5500	4400	2200	1100	550	220	110	44
MeOH Man./lot_____			1.0mL	1.0mL	1.0mL	2.0mL	2.0mL	2.0mL	10.0mL

Analyst:_____
Date:_____

QUALITY CONTROL TSC
Restek Q Unleaded Gasoline Composite Std. Lot_____ 20000ug/mL
QGRO=1:10 Restek Q Unleaded Gasoline Composite Std.

Low Level

Stock mix	QGRO _____	Final Volume	Prep Used
1000 ppb GRO	2.5 ul	5 ml Syringe	
1000 ppb GRO	25.0 ul	50 mL Flask	

Analyst:_____
Date:_____

High Level

		mL stock	Final Vol
QGRO :	2000 ug/mL	0.25	10mL
8260SS lot # :	2500 ug/mL	0.01	10mL
R826 SS :	25 ug/mL	1	10mL

Then dilute 1.0mL of spiked extract into a 50.0mL flask
Final Spike Concentration = 1000 ppb GRO

eurofins	Always check on-line for validity **Preparation of Vials for Field Preservation of Soils for Volatile Analysis**	Level: **Work Instruction**
Document number: **S-SS-WI11242**		
Old Reference: **1-P-QM-WI-9015073**		
Version: **16**		Organisation level: **5-Sub-BU**
Approved by: **UCSS** Effective Date **30-SEP-2016**	Document users: **5_EUUSLA_Sample Support_Manager, 6_EUUSLA_ Sample Support_VOA prep**	Responsible: **5_EUUSLA_Sample Support_Manager**

LIMS ID

Analysis DOD - 0388, 6119, 6169, 6647, 0405, 1169, 6171, 6172, 6173, 6645, 2392, 6176, 7579, 0069

Revision Log

Revision: 16		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Procedure	Reflects the current procedure	Referenced the option to use pre-purchased vials

Revision: 15		**Effective Date:** May 12, 2015
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version

Revision: 15		**Effective Date:** May 12, 2015
Throughout Document	Clarification	Replaced references of Parallax with LIMS
Cross Reference	1-P-QM-QMA-9017363 was made obsolete	Deleted 1-P-QM-QMA-9017363 and added 1-P-QM-QMA-9015389
Procedure	Reflects the current procedure.	Note section added to reference the use of purchased pre-tarred vials

Reference

1. Massachusetts Department of Environmental Protection Method for the Determination of Volatile Petroleum Hydrocarbons, May 2004.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 5035, November 2004.

3. Test Methods for Evaluating Solid Wastes, SW-846, Method 5035A, July 2002.

4. *Method for the Field Extraction/Preservation of Soil Samples with Methanol for Volatile Organic Compounds*, New Jersey DEP, February 1997.

5. Method AK101 for the Determination of Gasoline Range Organics, April 8, 2002.

6. *Instructions for EPA Reference Method 25D - Interlaboratory Comparison*, Research Triangle Institute, October 1991.

7. WI PVOC PUBL-SW-140 09/95, *Wisconsin DNR Modified GRO*.

8. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
1-P-QM-FOR-9008289	Field Preserved Vial Preparation for Volatile Soils
1-P-QM-PRO-9015516	Preservation and Bottles Room Preservative Traceability
1-P-QM-PRO-9015517	Pipette Dispenser Calibration Procedure
1-P-QM-PRO-9018271	Glassware Cleaning
1-P-QM-QMA-9015389	Balance, Syringe, Pipette Verification

Purpose
The purpose of this SOP is to provide detailed instructions for the preparation of vials used for field-preservation of soil samples to be analyzed for volatiles.

Scope
This procedure applies to analysts who prepare pre-preserved containers used in the field for soil sampling of volatile analyses.

Basic Principles
An aliquot of preservative is placed in a volatile-free container and weighed. The weight of the container and preservative is then captured using the Volatile Preparation program in LIMS. When requested, the container is sent to the client for use in field preservation of a solid sample. When the container and the soil sample are returned to the lab, it is re-weighed and the actual sample weight is calculated. Samples are then spiked with the appropriate surrogate as determined by the analysis being performed.

Definitions
1. Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD) – A sample of known composition analyzed with each batch of samples to demonstrate laboratory accuracy. The samples either naturally contain the analytes of interest or are clean samples fortified with known concentrations used to demonstrate laboratory accuracy. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

2. VOA Prep Summary and VOA Prep Summary by SDG are reports that reside on the LIMS database. Using the Volatile Prep application and moisture results pulled from LIMS, the report is populated and calculations are performed to achieve a Final Extraction volume to be used when necessary by the appropriate Groups.

Interferences
Sample contamination can occur if the vial preparation is not performed in a volatile-free environment; therefore, this process must be performed in one of the designated volatile-free laboratories. Samples also become contaminated if volatiles diffuse through the sample vial septum. A trip blank carried through sampling, storage and handling acts as a check of such contamination.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Methanol is flammable. Containers of this solvent must be kept away from any sources of open flames or sparks. Due to the potentially toxic nature of samples received from clients for analysis, safety precautions must be observed when handling samples. Safety glasses, lab coats, and gloves are required. Weighing out of the samples must always take place in a hood.

The solvents utilized in this procedure are disposed of in a solvent waste container which is transferred to the lab-wide disposal facility. Expired standards in methanol are disposed of as hazardous waste. Bulk sample containers and methanolic sample preparations are returned to the sample storage area for future disposal. Other wastes generated by Lancaster Labs are disposed of via incineration at EPA licensed facilities.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP.

The initial training consists of observing the procedure being carried out by an experienced analyst/technician. Next, the trainee performs the procedure while the experienced person watches, answers questions, and gives feedback. Following the initial training, experienced individuals are available as a resource until no longer required. Analysts are considered proficient when they are able to perform the procedure independently.

Sample Collection, Preservation, and Handling

Sample containers must be refrigerated at 0° to 6°C, not frozen, after preparation. Containers containing surrogates are not kept for more than 2 weeks before being discarded or sent into the field for use in sample collection. Packaging of sample containers must follow all DOT regulations. When returned, samples must be preserved and refrigerated unfrozen at 0° to 6°C or frozen in reagent water at −10° to −15°C within 48 hours of collection. See individual technical area SOPs for method specific hold times.

Apparatus and Equipment

1. 40-mL vials with Teflon™-lined septa and screw caps

2. 40-mL vials with stir bars, Teflon™-lined septa, and screw caps, SciSpec Catalog #376740-MB or equivalent

3. 125-mL amber glass wide mouth jar with Teflon™-lined septa and screw caps

4. Pipette capable of dispensing up to 25 ± 0.25 mL. Refer to 1-P-QM-PRO-9015517 (SOP-SS-018) for calibration procedures.

5. 50-µL syringe

6. 1000-mL volumetric flask, class A

7. Analytical balance capable of weighing ±0.01 g. Refer to 1-P-QM-QMA-9015389 (LOM-SOP-ES-235) for calibration procedures.

8. Label printer/labels

9. LIMS VOA prep application and Reagent, Solution, and Balance Applications which integrates a PC with an analytical balance to collect data directly from the balance. It organizes the data, performs calculations, and stores final results in the Laboratory Information Management System (LIMS).

Reagents and Standards

NOTE: A rinse using reagent water followed by methanol must be performed on the pipette dispensers before adding a new lot of solution or standards.

1. Methanol – Purge and trap grade.

   a. Store at room temperature and re-analyze yearly.

   b. Use methanol that has been previously tested and approved for use by the labs. See 1-P-QM-PRO-9015516 (SOP-SS-017) for further information.

2. 8260A/B Surrogate Mix, Restek Catalog #30340 (2500 µg/mL) or equivalent.

   a. Store at -10° to -15°C for up to 1 year.

   b. Use standard as is, or diluted in methanol to a final concentration of 2.5 µg/mL. Diluted standard must be stored at -10° to -15°C for up to 6 months.

c. This standard is used for the GC/MS analyses.

3. Custom α,α,α−trifluorotoluene (TFT), Restek Catalog #54357 (15,000 µg/mL) or equivalent.

a. Dilute standard in methanol to a final concentration of 750 µg/L for the working solution. A prep dilution of 0.5 mL of TFT to 10 mL of methanol is used when spiking returned field preserved containers.

b. Store at −10° to −15°C for up to 30 days.

c. This standard is used for the GC analyses.

4. Sodium hydrogen sulfate anhydrous powder, Fluka, Catalog #2316657 or equivalent.

a. Store at room temperature and re−analyze yearly.

b. If compounds are detected above the method detection limit (MDL), prepare another vial and repeat the analysis. If compounds are still detected above the MDL, a new container must be tested and used.

5. Sodium Bisulfate Solution –

a. Prepare by diluting 200 ±0.5 g of the Sodium hydrogen sulfate anhydrous into 1000 mL of reagent water in 1000−mL volumetric flask. Cap and invert at least 10 times to mix.

b. Store at room temperature and re−analyze every 6 months if supply remains.

c. If compounds are detected above the method detection limit (MDL), repeat the analysis. If compounds are still detected above the MDL, remake the solution and test before using.

6. Reagent water – water in which target analytes are not detected at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

7. Polyethylene glycol (PEG) – Average molecular weight 400 amu, EM Science preferred.

a. Any lot/vendor must meet a cleanliness level of <50 mg/kg volatile content.

b. Store at room temperature and re-analyze each year if supply remains.

Calibration

Not applicable to this procedure.

Procedure

Use the VOA Prep application whenever possible for this procedure to facilitate data transfers and other tracking. However, it is also allowable to record data traditionally in a logbook.

A. Prepare preservative containers

NOTE: Pre-purchased vials can be used for selected preps. Vials containing 5 mL of DI Water, vials containing 5 mL of Sodium Bisulfate and vials containing 5, 10 and 15 mL of methanol are available for field preserved analyses. The vials contain the initial tare weight, vial lot number, and the preservative lot number.

1. Check to make sure the pipette calibration has been performed.

2. Use the pipette to add the appropriate amount of preservative to a clean container (see 1-P-QM-FOR-9008289, Form 4580). A 40-mL vial is used unless otherwise indicated on Form 4580.

a. For analyses 6119 and 6172 the appropriately diluted surrogate solution must be added at this time using a pipette.

b. For analysis 1169 use a syringe to add the ampulated 8260A/B surrogate standard plus methanol into a 40-mL vial. Use 1 µL of 8260A/B surrogate for every 1 mL of methanol in the vial.

3. Seal the container with a screw cap and septum seal.

4. Label the container with the tracking number.

5. Check to make sure the balance has been calibrated each day before use.

6. Place the container on a zeroed balance and capture the weight electronically to the nearest 0.01 g using the VOA Prep application.

7. Store the prepared containers in the designated area in the Volatile Prep room. Vials containing surrogates must be stored in the refrigerator, vials containing just methanol are stored at room temperature or refrigerated.

8. Pull vials as requested via e-mail from client services and place on the designated shelf in the bottles room storage area. The containers are now ready to be sent into the field for sample collection.

9. Send a reply e-mail to the client service representative to notify them that the order has been completed.

B. Re-weigh preservative containers after return from the field

1. Using the Volatile Prep application, scan the tracking number on the container. This brings up the information documented from the preparation step described above.

NOTE: If using purchased pre-tarred vials insert the vial ID, preservative, and tare weight information on the Pre-Tarred tab within the Volatile Prep Application.

2. Check to make sure the balance has been calibrated each day before use. Place the container on a zeroed balance and capture the second weight electronically using the VOA Prep application.

3. The Volatile Prep application calculates the net weight. (weight of vial, solution, and soil minus the weight of the vial and solution).

4. If there are any holding-time issues or if weights are outside of the Action Requirement listed on 1-P-QM-FOR-9008289 (Form 4580), the VOA Prep application automatically sends an e-mail to Client Services.

5. Record any unusual observations about the sample in the comment section.

C. Sample spiking

NOTE: Analyses 11014 and 11764 do not require a surrogate to be spiked into the methanol.

A 1:1 ratio of µL to mL is required when spiking surrogate into methanol, except for analysis 0388 MA-VPH samples which require 30 µL to the 15 mL of methanol.

1. Determine the amount of methanol in the sample. This is determined by the analysis number and the mL of methanol printed on the tracking number.

2. Fill the syringe with the appropriate surrogate slightly past the amount of µL needed.

a. Add the appropriate surrogate based on the bottle code entered and the department receiving the sample.

b. GC requires a TFT surrogate mix to be used, bottle code 64.

c. GC/MS requires an 8260A/B surrogate mix to be used, bottle codes 14 and 66.

3. Dispense the surrogate from the syringe until the required level of µL is reached. This assures no air bubbles are present and the syringe contains surrogate.

a. Tilt the vial so the methanol comes toward the top of the vial, spike through the septa, submerging the tip of the syringe into the methanol.

b. Shake the vial to disperse the surrogate throughout the sample.

c. Put an “S” on the vial label to indicate surrogate has been added.

d. Clean the syringe according to 1-P-QM-PRO-9018271 (SOP-SS-026) before proceeding to the next sample.

D. Deliver samples to the labs

Once the sample containers have been re-weighed, they must be transported to the laboratory for analysis. Each department has a designated refrigerated drop-off spot. All trip blanks and field blanks submitted must follow the sample containing the same preservative. Blanks submitted as waters are to be stored in the designated volatile refrigerator.

NOTE: Any sample designated for prescreen must be taken to the appropriate refrigerator and placed in a box sorted in numerical order in groups of 21 or 42 when applicable. If a methanol sample is designated for prescreen and a corresponding methanol trip blank or field blank is submitted the blanks must follow the associated samples through the entire process including sample prescreen.

Calculations

Calculation of sample weight : Wn = Ws - Wf

Where:

Wf = weight of container + solution (first weight)

Ws = weight of container + solution + soil (second weight)

Wn = net weight of soil sample

Statistical Information/Method Performance

Not applicable to this procedure.

Quality Assurance/Quality Control

The number of containers requested from Client Services includes the appropriate amount of extra bottles to serve as matrix quality control (QC) for volatile analyses requested. The matrix QC spiking for this process is noted in Procedure C. All other QC samples such as the LCS, LCSD, and method blanks are prepared as outlined in the individual technical areas SOPs.

End of document

Version history

Version	Approval	Revision information
16	30.SEP.2016	

eurofins	Always check on-line for validity **Determination of Volatile Target Compounds and Gasoline Range Organics (GRO) by Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS) in Waters and Wastewaters by Method 8260C**	Level: **Work Instruction**
Document number: **T-VOA-WI8194**		
Old Reference: **1-P-QM-WI- 9013078**		
Version: **5**		Organisation level: **5-Sub-BU**
Approved by: **UCSS** Effective Date **29-JUN-2016**	Document users: **6_EUUSLA_GC/MS Volatiles_GC/MS Auditors/Verifiers, 6_EUUSLA_GC/MS Volatiles_Level II Peer Review, 6_EUUSLA_GC/MS Volatiles_Management, 6_EUUSLA_GC/MS Volatiles_SW846 8260B/C Water**	Responsible: **5_EUUSLA_GC/MS Volatiles_Manager**

LIMS ID
Analysis 11996, 11997, 13130

Revision Log

Revision: 5		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Cross Reference	Updated	Change document name for 1-P-QM-PRO-9015467
Entire Document	Continuity	Removed all unnecessary "To prevent confusion" statements
Reagents and Standards B.1.c.3	Requirement	Added preparer name and storage method to label requirements

Revision: 5		**Effective Date:** This version
Reagents and Standards B.2	Clarification Current practice	Adjusted wording concerning working standard documentation Added 1 ppb and 0.2 ppb levels to list of calibration levels
Calibration C.	Current practice	Changed number of calibration levels from 6 to 7
Attachment I	Current practice	Added C5 retention time marker information
Appendix	Enhancement	Replaced Figures 1-5

Revision: 4		**Effective Date:** Jan 22, 2016
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Scope	Clarification	Included disclaimer concerning TSC sheets
Personnel Training and Qualifications	Duplicate information	Removed the statement regarding the SOP and DOC at the beginning of the section.
Calibration C.2	Continuity	Moved (2) below calibration table
Calibration E.	Correction	Changed check standard concentration for 25ml purge analysis
Calibration F.	Enhancement	Added MDL sensitivity check requirement
Appendix	Updated with most current version	Replaced Figures 1-5

Reference

1. Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), SW-846 Method 8260C, August 2006.

2. Determinative Chromatographic Separations, SW-846 Method 8000B, December 1996.

3. Purge and Trap for Aqueous Samples, SW-846 Method 5030B, December 1996.

4. Purge and Trap for Aqueous Samples. SW-846 Method 5030C, Rev 3, May 2003.

5. *Total Petroleum Hydrocarbons Analysis-Gasoline Method*, California Department of Health Services, LUFT Task Force.

6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
1-P-QM-PRO-9015465	Glassware Cleaning
1-P-QM-PRO-9015467	GC and GC/MS Instrumentation Maintenance
1-P-QM-PRO-9015469	GC/MS Volatile Standards Traceability
1-P-QM-PRO-9015470	Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles
1-P-QM-PRO-9015471	GC/MS Volatiles Audit Process
1-P-QM-PRO-9017810	Level II Review of GS/MS Volatiles

Document	Document Title
1-P-QM-QMA-9015390	Demonstrations of Capability
1-P-QM-QMA-9017309	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is suitable for the determination of the target compounds listed and maintained in the LIMS (Laboratory Information Management System) for aqueous matrices. Associated MDLs/LOQs are also listed in the LIMS under the analysis number and/or Project Information lists. Non-target volatile compounds in the sample can be tentatively identified (TIC) using a mass spectral reference library comparison. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by purge and trap GC/MS methodologies and skilled in mass spectral interpretation. Using this method, the TICs are quantitated with an estimated concentration. Compounds other than those listed in the LIMS for this group of master scans are analyzed using USEPA SW-846 Method 8260C. Theoretical Standard Calibration (TSC) Sheets are included in the Appendix (Figures1-6). These TSC sheets are to serve as examples only and may not reflect most current version in use. Attachment I describes the proper analysis procedure for Gasoline Range Organics in Water. Due to poor purging efficiency or poor gas chromatographic performance, some analytes require calibration at higher levels and higher practical quantitation limits (PQLs). Any additional compounds must be added to the theoretical standard concentrations (TSC) sheet. Standards containing additional analytes must be prepared as described in the Standards section of this document. Both secondary stock solutions and matrix spike solutions must be prepared for use in analyzing additional compounds.

Basic Principles

A 5-mL or 25-mL sample or a dilution of a sample is placed in a specially designed purge vessel. The sample is purged with an inert gas and the effluent gas passed through a sorbent tube where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and backflushed on to the head of a gas chromatographic (GC) capillary column. The GC column is temperature programmed to separate the volatile compounds, which are subsequently detected and identified using mass spectrometric techniques.

When a compound reaches the Mass Spectrometer, it is bombarded by high-energy electrons (70 eV). This causes the compound to fragment and form ions. The positive ions are focused into a quadrupole mass analyzer, where the ions are separated according to their mass/charge ratios during rapid repetitive scans. These ions are then amplified and detected with an electron multiplier.

The resulting time/intensity/mass spectra data are stored and processed by a computer. Target compounds are identified on the basis of relative retention times and mass spectral matches to standards, which are injected every 12 hours on the same system. The internal standard method is used for quantitation.

Interferences

Contaminant sources are volatile compounds in the laboratory environment, impurities in the inert purging gas, carryover from samples containing high concentrations of volatile organic compounds and dirty glassware. The analyst must demonstrate that the system is free from interferences (by producing acceptable method blank data) before analyzing a batch of samples. Matrix effects from heavily contaminated waters can interfere with the internal standard responses, target analytes and surrogate recoveries, thereby hindering accurate quantitation. See Section 4.0 of SW−846 Method 8260C for further discussion.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as the use of fume hoods, safety glasses, lab coats, and gloves. Neat compound sources and stock solutions must be collected into a lab pack upon expiration. The lab pack is delivered to Safety personnel for appropriate disposal. Expired secondary standard solutions in methanol must be disposed of as solvent waste. Pour expired secondary standard solutions into the appropriate solvent waste collection container. Aqueous calibration standard mixes are disposed of as nonhazardous aqueous waste due to the low concentration. Samples with a pH ≤2 are taken to storage until disposal in an acid waste container.

Personnel Training and Qualifications

Education Requirement: A 4−year Baccalaureate Degree from an accredited College or University in one of the physical sciences and/or one to three years of relevant gas chromatography experience.

Analysts must be trained in the proper method of volatile organic sample preparation and analysis as determined by the supervisor(s). All training and education relating to volatile organic sample preparation and analysis must be documented by each analyst in his/her training record. All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP.

Specifically, each new chemist trains with an experienced chemist for the first 12 weeks depending on the individual and his/her previous experience. The first 12 weeks are spent working one−on−one with the trainer. This time is less if the new chemist has prior relevant experience in GC/MS and/or analytical chemistry background.

During the training period, the new chemist learns daily maintenance, calibration techniques, data and library search review, and forms generation. He/she is also required to read all relevant SOPs and EPA methods.

To evaluate the proficiency of each chemist, several checks have been established. The first is the ability to successfully calibrate. The chemist analyzes a series of at least five calibration standards and performs the calibration routine. Secondly, each analyst must perform a Demonstration of Capability (DOC). Refer to 1−P−QM−QMA−9015390 for specific requirements. Demonstration of Capability is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

The samples to be analyzed with this method must be stored in a refrigerator at 0°C to 6°C, not frozen. Samples are collected in 40−mL vials with no headspace. Preserve samples to a pH of <2 in order to prevent degradation of aromatic compounds that are present in the sample. 1:1 HCL is the recommended preservative. Preserved samples must be analyzed within 14 days of collection; those that are not preserved must be analyzed within 7 days of collection.

Apparatus and Equipment

1. Gastight micro-syringes – 1 to 1000 µL (various sizes)

2. 5−mL gastight syringes

3. Analytical balance, capable of accurately weighing ±0.0001 g

4. Glassware

   a. Class-A Volumetric flasks with ground-glass stopper

   b. Vials, 1.5-mL, 15-mL, and 40-mL screw cap, with Teflon™/silicone septa

   c. Mininert vials, 1 mL, 2 mL, and 5 mL

5. Purge and trap device – Consisting of the sample purger, the trap, and desorber; the OI Analytical 4560, OI Analytical 4660, or equivalent meets the requirements of this method. The purging

chamber must have the purge gas passing through the sample as finely divided bubbles and minimize the headspace between the sample and the trap to <15 mL.

6. Autosampler – OI Analytical 4551, OI Analytical 4552, Archon, or equivalent meets the requirements of this method.

7. Spiker unit – OI analytical Model 4551/4552 SAM/Spiker or equivalent. One or two automated syringe spikers can be added to the OI Analytical Model 4551/4552 autosampler to automatically introduce 1 µL of internal standard (ISTD), surrogate standard, and/or matrix spiking solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of appropriate standards.

8. GC/MS system – The Agilent 5890GC/5972 MSD, Agilent 6890GC/5973MSD, Agilent 6890GC/5975MSD and Shimadzu GC/MS QP5000 meet the requirements for this method.

9. Data System/Computer/Software – this is interfaced to the GC/MS system that continuously acquires and stores data during the analysis, and can process/reduce data to generate the appropriate forms and supporting data. The software used for acquisition is HP Chemstation®, and data reduction is accomplished using Target® software.

10. GC Columns

a. Column 1 – 30M × 0.25 mm ID DB624 capillary column with a 1.4−µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

b. Column 2 – 20M × 0.18 mm ID DB624 capillary column with a 1.0−µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

c. Column 3 – 20M × 0.18 mm ID DB-VRX capillary column with a 1.0−µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

NOTE: Refer to 1−P−QM−PRO−9015467 for instrumentation maintenance and troubleshooting.

Reagents and Standards

A. Reagents

1. Reagent water is defined as water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets these criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Methanol, Purge and Trap Grade or equivalent.

B. Standards

See 1-P-QM-PRO-9015469 for standards traceability.

1. Stock standard solutions – Stock solutions must be prepared in methanol. Standards are prepared from ampulated and neat compounds obtained from suppliers that indicate the purity of the compound. No correction for purity is made if the purity is listed as ≥96%. Pre-made solutions can be used if the supplier documents the concentrations of the solutions. All ampulated standards are stored at −10° to −15°C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided.

a. For most of the target compounds, the stock standard solutions are purchased from a vendor as custom mixes (V for calibration and Q for separate source quality control). The internal and surrogate standards are purchased from a vendor, as well as the target compounds that are gases at room temperature. These gaseous standards have a 1-week expiration date, starting from the date they are opened.

b. 8260A Surrogate standard spiking solution (8260SS) – a 2500 µg/mL stock standard solution of dibromofluoromethane, toluene-d8, 4-bromofluorobenzene, and 1,2-dichloroethane-d4 is prepared in methanol by a commercial supplier.

c. 8260A Internal standard spiking solution (8260IS) – a 2500 µg/mL stock standard solution of fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, and 12500 µg/mL deuterated tertiary butyl alcohol (tBA-d10) is prepared in methanol by a commercial supplier. Deuterated tertiary butyl alcohol (tBA-d10) is used sometimes as an auxiliary ISTD.

To prepare stock standards from neat compounds:

(1) Place about 9.8 mL methanol or an equivalent solvent into a tared 10.0-mL glass-stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

(2) Add the liquids using a syringe or pipette by adding 2 or more drops of the assayed material to the flask, being careful that no drop hits the side of the flask. Reweigh the flask, record/note the amount, dilute to volume, stopper, and mix by inverting the flask at least 3 times. Calculate the concentration of the standard.

(3) The stock standard solutions are stored in Teflon™-sealed screw-capped vials at −10° to −15°C. The compound name, concentration, date prepared, expiration date, preparer name and storage method must appear on the bottle.

(4) Replace in-house prepared stock standard solutions every 6 months.

2. Secondary dilution standards – Using the stock standard solutions, prepare secondary stock solutions in methanol containing the desired compounds. These standards are prepared by calculating the volume of each stock standard required to produce a given volume of a mixed working standard with

a known concentration of each analyte. When custom mixes are used, these are diluted down individually or combined together with other mixes. The working standard is tested according to 1−P−QM−PRO−9015469. The verified working standard is poured into Teflon-lined screw−capped GC vials or mininert vials and stored at −10° to −15°C. A designator indicating the standard name, month, and day of preparation and expiration date must be on the standard vials. The designator and all data pertaining to the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every 6 months unless otherwise indicated.

a. 1,4-Bromofluorobenzene (BFB) standard – Prepare a 50−µg/mL solution of BFB in methanol by diluting the stock standard (prepared from neat material) with methanol to a final volume of 100 mL. The volume of stock standard used varies depending on the actual stock concentration.

b. IS/SS spiking solution – Dilute 1 mL of 8260IS and 1 mL of 8260SS with methanol to 10−mL final volume (resulting in a concentration of 250 µg/mL, 1250 µg/mL for tBA-d10). This is assuming a 1−µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained.

c. Calibration spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. Suggested calibration levels are 1, 4, 10, 20, 50, 100, and 300 ppb for 5−mL purge analysis. Suggested calibration levels are 0.2, 0.5, 1, 2, 5, 10, and 25 ppb for 25−mL purge analysis. A Theoretical Standard Concentration (TSC) sheet is filled out for all initial calibrations (see Figures 1 and 2). Replace calibration spiking solution every month.

d. Matrix spiking solution – Prepare second source solutions in methanol that contain the compounds of interest at known concentrations.A TSC sheet is filled out for all quality control samples (see Figures 4 and 5). These solutions serve as both the matrix spiking solution and the laboratory control sample solutions. Matrix spikes also serve as duplicates. Therefore, two aliquots of the same sample need to be spiked for analysis with these solutions. Replace matrix spiking solution every month.

Store all standard solutions at −10° to −15°C.

Preparation of Glassware

All glassware is cleaned according to 1−P−QM−PRO−9015465.

Calibration

A. Instrument conditions

1. The purge and trap device must have the trap conditioned for at least 10 minutes at 180° to 220°C at a flow rate of 20 to 60 mL/min prior to initial use.

2. An example of purge and trap conditions are listed below:

Purge Gas	Helium
Purge Flow	35 - 45 mL/min
Purge Temperature	40°C for 8260C waters
Purge Time	11 minutes
Desorb Temperature	190°- 220°C
Desorb Time	0.5 to 4 minutes **
Bake Temperature	180°-220°C
Bake Time	5 – 16 min

**Range as suggested by the purge and trap instrument manufacturer

NOTE: Purge and trap conditions are changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument is found in the appropriate instrument maintenance log.

3. The suggested gas chromatographic operating conditions are listed in the table below, depending on the column used:

	Column 1	1 *Column 2*	2 *Column 3*
Column liquid phase	DB-624	DB-624	DB-VRX
Carrier gas	Helium	Helium	Helium
Carrier gas flow	0.8 mL/min	0.6 mL/min	0.6 mL/min
Make-up gas flow	None	None	None
Initial temperature	45°C	45°C	45°C
Initial hold time	4.5 min	2.5 min	4 min
Temperature ramp	12°/min until 100°C then 25°/min until 240°C	12°/min until 100°C then 25°/min until 235°C	25°/min until 60°C then 36°/min until 240°C
Final temperature	240°C	235°C	240°C
Final hold time	None	.02 min	1 min

4. The recommended mass spectrometer operating conditions are listed below:

Mass range:	35 – 300 amu
Scan time:	One scan cycle per second or less and resulting in at least five scans per chromatographic peak

NOTE: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions that give the performance required by the method are acceptable.

B. Tuning

Tune the GC/MS system to meet the criteria in Table 1 following a 50−ng injection of BFB. The chromatographic conditions must be the same as those under which the samples are analyzed except that the temperature ramp is increased and the initial temperature and flow rate is different. The BFB tune must be verified every 12 hours.

The tune must be evaluated by taking the average of the three scans across the BFB peak apex with a background subtraction of a scan within 20 scans prior to the start of the BFB peak.

NOTE: All standards, samples, and associated quality control samples must be analyzed with the same MS parameters as those used to obtain a successful tune.

C. Initial calibration

1. The initial internal standard calibration consists of analyzing seven distinct levels of analyte concentrations and producing response factors for each compound (six levels are required if second order regression fits are used). Refer to Figure 1 or 2 for the preparation of the calibration standards.

The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of the analyte concentration.

NOTE: 5 levels of standard are required by the method.

a. When using an OI 4552 or OI 4551 autosampler, the standards (including target and surrogate compounds) are prepared and poured into 40−mL vials with Teflon™−lined septa. A 5−mL or 25−mL aliquot is withdrawn from the vial by the autosampler. The aliquot is transferred through the spiker unit to add the IS/SS spiking solution and then transferred to the sparge vessel.

b. Purge and desorb according to Calibration A.

c. Collect GC/MS data until the end of the GC run.

d. Empty and rinse the purging chamber at least twice with reagent water prior to loading another sample into the vessel, to minimize the possibility of carryover contamination.

e. Each level is analyzed as described above. Next, tabulate the area response of the characteristic ions (Table 2) against concentration for each analyte, surrogate standard, and internal standard and calculate relative response factors (RRF) for each compound (see Calculation section). The calibration is valid for 12 hours from the injection of the BFB tune standard, at which time a new

tune check and a continuing calibration check standard are evaluated prior to the analysis of additional samples. The following table describes the guidelines for an acceptable initial calibration:

Frequency	Acceptance Criteria	Corrective Action
Initially and then when analytes in the daily calibration standard fail criteria.	1. % RSD of ≤20% is required for all analytes. 10% of the analytes may fail this criteria. 2. All compounds of interest must be detected in the MDL standard. 3. The relative retention times of the target compounds must agree within 0.06 relative retention time (RRT) units. The exception would be in the case of system maintenance. 4. Minimum response factors must be met for select compounds. See Table 3.	1. Any target analyte with a %RSD of ≤20% must use the average RRF for quantitation. For any analyte in which the %RSD >20%, a first-degree linear regression can be used (providing that the correlation coefficient [CC] is ≥0.99). A quadratic fit ** (using 6 stds) can also be used (provided the coefficient of determination [CD] is ≥0.99). If the linear fit and quadratic fit pass the criteria for any given analyte, then use the line/curve with the smallest positive y-intercept. If the y−intercept quantifies to be greater than the LOQ, consult your supervisor immediately or recalibrate. If CC or CD is <0.99, recalibrate. Supervisory approval is required for exceptions to these guidelines. If >10% target analytes fail, recalibration is required. 2. If a compound is not detected in the MDL standard, then report to the level of the lowest standard detected. 3.-4. Perform system maintenance and recalibrate.

**Consult USEPA method 8000B for non-linear curve fitting techniques/guidelines

NOTE: If a linear fit is used for a compound, the lowest calibration standard point must be recalculated against the curve. The recalculated concentration must be within ± 30% of the standard's true concentration. If this criteria is not met, notify a supervisor so that an alternate LOQ can be evaluated.

2. A method detection limit (MDL) standard must be analyzed with each initial calibration. This standard is prepared at or near the departmental MDL and is not to be included in the calibration curve. All compounds must be detected in the MDL standard. (See Figure 1 or 2 for the preparation information).

D. Following the calibration, an Initial Calibration Verification (ICV) standard must be run. The ICV is prepared according to the TSC sheet in Figures 3 and 5. The ICV acts as a second source standard to check against the initial calibration. All analytes must meet ICV acceptance windows of 70%-130%. If

the ICV does not meet the aforementioned criteria, a second ICV is analyzed before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

E. Continuing calibration verification (CCV) – The CCV is performed by analyzing a CCV standard in subsequent tune periods after an initial calibration. The CCV is analyzed at 50 ppb for 5−mL purge waters and 10 ppb for 25−mL purge waters. The CCV is considered valid when the criteria listed below are met:

Frequency	Acceptance Criteria	Corrective Action
Every 12 hours.	1. % Drift of ≤20% is required for all analytes. 20% of analytes may fail this criteria if not detected in proceeding samples. 2. The relative retention times (RRT) of the target compounds must agree within 0.06 RRT units. The exception would be in the case of system maintenance. 3. The extracted ion current profile (EICP) area for each internal standard must fall within the window of –50 % to +100 % from the mid-level standard area produced during the last initial calibration. 4. Minimum response factors must be met for select compounds. See Table 3.	1.-4. In the event that the continuing calibration verification (CCV) standard fails any of these criteria, sample analysis must be suspended and the CCV must be re-analyzed. If the re-analysis fails any of the criteria then adjustments are to be made to the analytical system to return it to its original condition, followed by the analyses of 2 consecutive CCVs at the same level that failed. If both CCVs pass the criteria, then sample analysis can continue. Otherwise, the system must be recalibrated and the samples reanalyzed, or the data can be qualified.

F. MDL Sensitivity Check- A MDL Sensitivity Check must be analyzed in cases where compounds fail to meet the % drift criteria in the CCV and have decreased sensitivity (-20% drift or greater). Affected compounds can be reported as non-detects if it is demonstrated that there is adequate sensitivity to detect the compound at the MDL. If the failed compound is detected, the concentration must be reported as an estimated value.

Procedure

A. Method Blank

Analyze the method blank as described above for the initial calibration standards. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than the reporting limit for the

associated samples. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met.

B. Laboratory Control Sample/ Duplicate and Matrix Spike/Duplicate: Refer to table in QA/QC section for specific requirements.

C. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The compound relative retention time must compare within ±0.06 RRT units of the RRT of the standard.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst discretion is used to determine compound identification. (Example: for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

5. The above criteria apply to hits greater than or equal to the LOQ. For hits between the MDL and the LOQ, both the criteria listed above and the analyst's discretion is used to determine compound identification.

6. The analyst must account for peaks that are greater than 10% relative intensity in the sample mass spectrum, but not present in the standard mass spectrum. Also, if a compound fails any of the criteria listed above but in the judgment of the mass spectral interpretation specialist is a correct identification, the identification is used and the quantitation of the peak is performed.

The primary and secondary ions for the target compounds can be found in Table 2.

D. Quantitative analysis

Once a compound has been identified, quantitation is based on the internal standard technique and the integrated area from the extracted ion current profile (EICP) of the primary characteristic ion. The list of primary characteristic ions is listed in Table 2.

E. Sample Analysis

A 5−mL or 25−mL aliquot of the sample is analyzed using the same instrumental conditions as the standard (whether ICAL or CCV), tune and method blank. If the QA criteria are satisfied and no target compounds are detected at concentrations above the calibration range, the results can be reported. To avoid possible matrix effects, sample carryover and re-analyses, an initial dilution is performed if:

1. Prescreening indicates a high volatile organic content in the sample

2. Historical data (or lack thereof) and/or sample appearance indicate a need for dilution

If target compounds are detected in the sample at concentrations above the calibration range, a dilution must be performed (See 1−P−QM−PRO−9015470 for information on when cleaning blanks must be run). See Section 11.5.6 in method SW-846 8260C for recommended dilution procedures.

Calculations

A. Calibration calculations

1. Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Characteristic ion area for the compound being measured

A(is) = Characteristic ion area for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression Equations:

1st Order (linear) regression: $Y = MX + B$

2nd order (quadratic) regression: $Y = CX^2 + MX + B$

Where:

x = Area(Std) /Area(Istd)

Y = Conc.(Std)/Conc.(Istd)

M = 1st degree slope

C = 2nd degree slope

B = Y-intercept

3. Percent relative standard deviation (%RSD):

$$\%\ RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

4. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

C(c) = Measured concentration using selected quantification method

B. QA Calculations

1. Calculation of percent recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

2. Relative percent difference (RPD):

$$RPD = \frac{MSR - MSDR}{(1/2)\ (MSR + MSDR)} \times 100$$

Where:

MSR = Matrix spike measured concentration

MSDR = Matrix spike duplicate measured concentration

3. Analyte Concentration

$$Concentration\ (\mu g/L) = \frac{(Ax)\,(Is)}{(Ais)\,(RRF)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Concentration of internal standard added in µg/L

RRF = Relative response factor from the initial calibration

Statistical Information/method Performance

The LCS must contain 80% to100% of the compounds in the calibration mix. LCS, MS, and surrogate recoveries and RPD are compared to the limits stored on the LIMS. These limits are statistically derived but must fall within 70% to 130% recovery for South Carolina compliance samples. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to 1–P–QM–QMA–9017309 for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

Each analysis batch (consisting of no more that 20 samples) must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD), a laboratory control sample/laboratory control sample duplicate (LCS/LCSD) or a duplicate (DUP). The LCS serves as a 2nd source standard verification of the initial calibration (ICAL). Additional QC samples are required to meet project or state certification requirements. Every sample or QC analysis must contain internal standards and surrogate compounds at a concentration of 50 µg/L for a 5–mL purge or 10 µg/L for a 25–mL purge.

Quality Control Item	Acceptance Criteria	Corrective Action
1 Internal Standards Added to every sample, standard, method blank and QC sample	1. *Peak areas within -50% to +100% of the area in the associated reference standard.* 2. Retention time (RT) within 30 seconds of RT for	1. Check instrument for possible problems and then reanalyze samples. 2. If re-injecting meets the criteria, report this analysis. 3. If this reanalysis still shows the same problem, report results from first analysis and qualify data with a comment.

Quality Control Item	Acceptance Criteria	Corrective Action
	associated reference standard.	
2 Surrogates Added to every sample, standard, method blank and QC sample	All % recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis.	If non-compliant, check for calculation or preparation errors. If no errors are found, check system for problems and reanalyze. If this reanalysis still shows the same problem, report first analysis and qualify data with a comment. If recoveries are outside of specification high and no target compounds are detected, then a reanalysis or comment is not required.
3 Method Blank (MB) Performed during each tune period after the initial calibration or CCV (minimum of 1 MB per 20 samples)	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. Quantitative results for all target compounds must be less than the reporting limit for the associated samples.	1.-2.Inspect system for possible problems and reanalyze. 3. If the MB contains target analytes and the associated samples do not, then no corrective action is required. If the target compounds in the MB are also in the associated samples, then they must be reanalyzed after a clean MB is obtained (certain projects may allow some exceptions for common laboratory contaminants like methylene chloride and acetone up to 5X the LOQ)
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) LCS analyzed with each batch of ≤ 20 samples LCSD analyzed if MS/MSD unavailable See Figures 4 and 5 for preparation info.	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All % recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis.	1.-2. If non-compliant, check for calculation or preparation errors. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD re-analysis still fails, perform appropriate system maintenance and restart the tune period. Only with a LCS % recovery failing high (for the requested target compounds) with targets non-detected in the sample, can the results be reported. Otherwise, the sample must be analyzed with a compliant LCS.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) MS/MSD analyzed with each batch of ≤ 20 samples (if sufficient sample volume available) See Figures 4 and 5 for preparation info.	1. % Recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries and/or RPDs outside of QC limits, consult the supervisor.

NOTE: Prior to release from the analytical laboratory, all data is reviewed in accordance with 1-P-QM-PRO-9015471 or 1-P-QM-PRO-9017810 (dual purge and trap).

Attachment 1

Gasoline Range Organics (GRO) by Gas Chromatography/Mass Spectroscopy (GC/MS)

This section is specific to the steps required for GRO analysis. See the main body of the SOP for general information/ processes.

Basic Principles:

The GRO analysis is typically performed in conjunction with the analysis of other volatile target compounds by SW-846 Method 8260C. The GRO quantitation range is 0.1 minutes before the peak apex of C6 (hexane) to 0.2 minutes after the peak apex of C12 (dodecane); however, other ranges can be established. By establishing a (C12) GRO window to 0.2 minutes following the elution of dodecane, the areas from a trio of unresolved peaks eluting near to the upper limit of the range must consistently be included in the total GRO area. In addition, the range remains tight enough to ensure that no C13 or greater compounds can be included in the total GRO area. The C4 range retention time is determined by selecting the first peak after the air and/or artifact peak minus 0.1 minutes in the first standard analyzed in the ICAL. The C5 range retention time is 0.1 minutes before the peak apex of pentane. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by GC/MS purge and trap methodologies. The area of the total ion chromatogram for the GRO range is determined. The area of the internal standards and surrogate standards are found and subtracted from the total area of the chromatogram within the desired time range. The resulting area is then quantitated versus the internal standard, fluorobenzene.

Interferences:

See main body of SOP.

Safety Precautions and Waste Handling:

See main body of SOP.

Personnel Training and Qualifications:

See main body of SOP.

NOTE: A separate Demonstration of Capability for GRO is required.

Sample Collection, Preservation, and Handling:

See main body of SOP.

Apparatus and Equipment:

See main body of SOP.

Reagents and Standards:

A. Reagents- See main body of SOP.

B. Standards- See main body of SOP for general standards.

1. GRO calibration standard – a 5500−µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier.

2. GRO QC standard – a 20,000−µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier

Store all standard solutions at −10° to −15°C

Calibration:

A. Initial calibration:

Prior to the analysis of any calibration level, retention time markers must be run for the GRO range of interest. The retention time markers are hexane (C6) and Dodecane (C12). Other markers can be used if different ranges are required by a project.

Internal standard calibration for GRO consists of analyzing six distinct levels of GRO area in order to produce a response factor for the GRO quantitation range of interest using the internal standard, fluorobenzene. The relative standard deviation of the response factor determines the suitability of the average relative response factor for calculation of the GRO concentration.

NOTE: 5 levels of standard are required by the method.

1. Prepare the calibration standards at appropriate levels. Suggested calibration levels are 44, 110, 550, 1100, 2200, and 4400 ppb.

To assure proper calibration, a Theoretical Standard Concentration (TSC) sheet is completed for each calibration (Figure 6). The TSC sheet contains the theoretical concentration for each certified analyte in the calibration at the various levels.

2. Each level is analyzed as described in the procedure under data analysis. Next, tabulate the area response for the GRO quantitation range minus the peak areas for the internal and surrogate standards that elute within the GRO range. Calculate the relative response factor (RRF) for GRO (see Calculation section) using the internal standard peak area for fluorobenzene.

NOTE: Although four internal standard compounds are spiked for the 8260B analysis, only one, fluorobenzene, is used for the quantitation of the GRO result.

3. Calculate the average relative response factors for the GRO quantitation range of interest. The calibration levels are evaluated on the basis of the relative standard deviation of the RRF values (% RSD). The %RSD for the GRO range of interest must be ≤20%. If the calibration meets this requirement then the average RRF is used to calculate sample concentrations. If the %RSD is >20% then re−analysis of one or more levels can be necessary before the calibration is valid.

B. Initial Calibration Verification (ICV):

Following the calibration, an Initial Calibration Verification (ICV) standard must be run. The ICV is prepared according to the TSC sheet in Figure 6 (QC prep). The ICV acts as a second source standard to check against the initial calibration. Results must quantitate within the 70-130% window. If the ICV does not meet the aforementioned criteria, a second ICV can be run before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

C. Continuing calibration verification (CCV):

The CCV involves an analysis for the 1100-ppb standard. The calibration is considered valid if the percent drift is ≤20%. Also, the internal standard peak area of fluorobenzene for the CCV is monitored against the mid-point standard of the initial calibration and must be –50% to +100% of the area counts. If any criteria listed above fails, the CCV is considered invalid. In the case where two consecutive CCVs fail, corrective action must be taken which can include re−analysis of the calibration check, instrument maintenance, and/or recalibration. If the criteria are met, the selected quantitation method from the initial calibration is used for blank and sample calculations until the end of the 12-hour period.

Procedure:

Samples must be analyzed in accordance with the analyses listed in the main body of this SOP. However, additional requirements are required for the GRO data analysis.

A. The Total Ion Chromatogram (TIC) is reviewed to insure proper integration around the 8260 surrogates and internal standards. Also the TIC is checked to make sure all major peaks are integrated.

B. The quantitation of the GRO range is performed using the equations listed in the Calculations section of this procedure. All calculations must report concentrations in values of µg/L. In the case where the

total GRO concentration exceeds the calibration range, the sample is re−analyzed at a dilution that brings the GRO concentration within the calibration range of the GC/MS system.

Calculations:

See main body of SOP.

Statistical Information/Method Performance:

See main body of SOP.

Quality Assurance/Quality Control:

See main body of SOP.

Table 1

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	base peak, 100% relative abundance
96	5% to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5% to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table 2

Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
trans-1,2-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77, 110
trans-1,3-Dichloropropene	75	77, 110
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzen-d4	152	115

Table 3
Minimum Relative Response Factors For ICAL and CCV

Volatile Compounds	Minimum Response Factor
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon Disulfide	0.100
Methyl Acetate	0.100
Methylene Chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100

Table 3 - Continued

Volatile Compounds	Minimum Response Factor
m&p-Xylene	0.100
o-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300

1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Attachment:
Figure 1

End of document

Version history

Version	Approval	Revision information
5	29.JUN.2016	

Figure 1

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
EPA SW846 Method 8260A/B/C

Date:_____
Instrument:_____

VOA1= 1:5 dilution of VCS#1B, VCS#2B, and VCS#4C
VOA2= 1:5 dilution of VCS#2B
VOA3= 1:5 dilution of VCS#3B and Vacrolein
VOA6= 1:5 dilution of VCS#6
2CEVE= 1:5 dilution of VCS#1B-2CEVE
VOA8= 1:5 dilution of Hexachloroethane and 2,2'-oxybis(1-Chloropropane)

Stock mix name	VOA1 _____ 2CEVE _____ 1,3-BUT _____	VOA3 _____	VOA2 _____	VOA6 _____ EE _____ Custom V LG Freons _____	n-PEN _____ VOA8 _____	CYC _____	EOH _____	Restek Gases (2000 ppm) Lt#_____ TAEE _____		Flask mL	
300 ppb std	15 µL	6 µL		15µL	15 µL	30 µL	30 µL	7.5 µL		50	
100 ppb std	5 µL	2 µL		5 µL	5 µL	10 µL	10 µL	2.5 µL		50	
50 ppb std	5 µL	2 µL		5 µL	5 µL	10 µL	10 µL	2.5 µL		100	
20 ppb std	4 µL	1.6 µL	4 µL	4 µL	4 µL	16 µL	16 µL	2.0 µL		200	
10 ppb std	2 µL	0.8 µL	2 µL	2 µL	2 µL	8 µL	8 µL	1.0 µL		200	
4 ppb std	4 µL	1.6 µL	12 µL	4 µL	4 µL	32 µL	20 µL	2.0 µL		1000 *	
1 ppb std	* Aliquot 12.5 mL of 1000 mL flask into 50 mL flask										
0.5 ppb MDL std	+ Aliquot 12.5 mL of 1000 mL flask into 100 mL flask										

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb	0.5 ppb
Benzene	CS#1B	5000	300	100	50	20	10	4	1	0.5
Bromobenzene		5000	300	100	50	20	10	4	1	0.5
Bromodichloromethane		5000	300	100	50	20	10	4	1	0.5
Bromoform		5000	300	100	50	20	10	4	1	0.5
n-Butylbenzene		5000	300	100	50	20	10	4	1	0.5
sec-Butylbenzene		5000	300	100	50	20	10	4	1	0.5
tert-Butylbenzene		5000	300	100	50	20	10	4	1	0.5
Carbon Tetrachloride		5000	300	100	50	20	10	4	1	0.5
Chlorobenzene		5000	300	100	50	20	10	4	1	0.5
Chloroform		5000	300	100	50	20	10	4	1	0.5
2-Chlorotoluene		5000	300	100	50	20	10	4	1	0.5
4-Chlorotoluene		5000	300	100	50	20	10	4	1	0.5
Dibromochloromethane		5000	300	100	50	20	10	4	1	0.5
1,2-Dibromo-3-chloropropane		5000	300	100	50	20	10	4	1	0.5
1,2-Dibromoethane (EDB)		5000	300	100	50	20	10	4	1	0.5
Dibromomethane		5000	300	100	50	20	10	4	1	0.5
1,2-Dichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,3-Dichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,4-Dichlorobenzene		5000	300	100	50	20	10	4	1	0.5

Figure 1 Continued

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
EPA SW846 Method 8260A/B/C

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb	0.5 ppb
1,1-Dichloroethane	CS#1B	5000	300	100	50	20	10	4	1	0.5
1,2-Dichloroethane		5000	300	100	50	20	10	4	1	0.5
1,1-Dichloroethene		5000	300	100	50	20	10	4	1	0.5
cis-1,2-Dichloroethene		5000	300	100	50	20	10	4	1	0.5
trans-1,2-Dichloroethene		5000	300	100	50	20	10	4	1	0.5
1,2-Dichloropropane		5000	300	100	50	20	10	4	1	0.5
1,3-Dichloropropane		5000	300	100	50	20	10	4	1	0.5
2,2-Dichloropropane		5000	300	100	50	20	10	4	1	0.5
1,1-Dichloropropene		5000	300	100	50	20	10	4	1	0.5
cis-1,3-Dichloropropene		5000	300	100	50	20	10	4	1	0.5
trans-1,3-Dichloropropene		5000	300	100	50	20	10	4	1	0.5
Ethylbenzene		5000	300	100	50	20	10	4	1	0.5
Hexachlorobutadiene		5000	300	100	50	20	10	4	1	0.5
Isopropylbenzene		5000	300	100	50	20	10	4	1	0.5
p-Isopropyltoluene		5000	300	100	50	20	10	4	1	0.5
Methylene Chloride		5000	300	100	50	20	10	4	1	0.5
Naphthalene		5000	300	100	50	20	10	4	1	0.5
n-Propylbenzene		5000	300	100	50	20	10	4	1	0.5
Styrene		5000	300	100	50	20	10	4	1	0.5
1,1,1,2-Tetrachloroethane		5000	300	100	50	20	10	4	1	0.5
1,1,2,2-Tetrachloroethane		5000	300	100	50	20	10	4	1	0.5
Tetrachloroethene		5000	300	100	50	20	10	4	1	0.5
Toluene		5000	300	100	50	20	10	4	1	0.5
1,2,3-Trichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,2,4-Trichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,3,5-Trichlorobenzene		5000	300	100	50	20	10	4	1	0.5
1,1,1-Trichloroethane		5000	300	100	50	20	10	4	1	0.5
1,1,2-Trichloroethane		5000	300	100	50	20	10	4	1	0.5
Trichloroethene		5000	300	100	50	20	10	4	1	0.5
1,2,3-Trichloropropane		5000	300	100	50	20	10	4	1	0.5
1,2,4-Trimethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,3,5-Trimethylbenzene		5000	300	100	50	20	10	4	1	0.5
m-Xylene		5000	300	100	50	20	10	4	1	0.5
o-Xylene		5000	300	100	50	20	10	4	1	0.5
p-Xylene		5000	300	100	50	20	10	4	1	0.5
1-Chlorohexane		5000	300	100	50	20	10	4	1	0.5
Pentachloroethane	CS#6	5000	300	100	50	20	10	4	1	0.5
Allyl Chloride		5000	300	100	50	20	10	4	1	0.5
Bromochloromethane		5000	300	100	50	20	10	4	1	0.5
Methyl Acetate		5000	300	100	50	20	10	4	1	0.5
Methylcyclohexane		5000	300	100	50	20	10	4	1	0.5
2-Methylnaphthalene		5000	300	100	50	20	10	4	1	0.5
1,2,3-Trimethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,2-Diethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,3-Diethylbenzene		5000	300	100	50	20	10	4	1	0.5
1,4-Diethylbenzene		5000	300	100	50	20	10	4	1	0.5

Figure 1 Continued

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
EPA SW846 Method 8260A/B/C

Compound name	Std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb	0.5 ppb
Methacrylonitrile	CS#2B	12500	750	250	125	100	50	40	10	5
Propionitrile		25000	1500	500	250	200	100	80	20	10
trans-1,4-Dichloro-2-Butene		12500	750	250	125	100	50	40	10	5
t-Butyl Alcohol		25000	1500	500	250	200	100	80	20	10
2-Propanol		25000	1500	500	250	200	100	80	20	10
Isobutyl Alcohol		62500	3750	1250	625	500	250	200	50	25
n-Butanol		125000	7500	2500	1250	1000	500	400	100	50
1,4-Dioxane		62500	3750	1250	625	500	250	200	50	25
2-Butanone	CS#3B	25000	600	200	100	40	20	8	2	1
2-Hexanone		25000	600	200	100	40	20	8	2	1
4-Methyl-2-Pentanone		25000	600	200	100	40	20	8	2	1
Acetone		25000	600	200	100	40	20	8	2	1
Acrylonitrile		12500	300	100	50	20	10	4	1	0.5
2-Nitropropane		25000	600	200	100	40	20	8	2	1
Tetrahydrofuran		25000	600	200	100	40	20	8	2	1
Methyl-t-butyl Ether	CS#4C	5000	300	100	50	20	10	4	1	0.5
Ethyl Methacrylate		5000	300	100	50	20	10	4	1	0.5
Methyl Methacrylate		5000	300	100	50	20	10	4	1	0.5
Freon 113		5000	300	100	50	20	10	4	1	0.5
Hexane		5000	300	100	50	20	10	4	1	0.5
Heptane		5000	300	100	50	20	10	4	1	0.5
Cyclohexane		5000	300	100	50	20	10	4	1	0.5
Benzyl Chloride		5000	300	100	50	20	10	4	1	0.5
Methyl Iodide		5000	300	100	50	20	10	4	1	0.5
Carbon Disulfide		5000	300	100	50	20	10	4	1	0.5
2-Chloro-1,3-Butadiene		5000	300	100	50	20	10	4	1	0.5
di-Isopropyl Ether		5000	300	100	50	20	10	4	1	0.5
tert-Amyl Methyl Ether		5000	300	100	50	20	10	4	1	0.5
Ethyl-t-butyl Ether		5000	300	100	50	20	10	4	1	0.5
Bromomethane	Gas mix	2000	300	100	50	20	10	4	1	0.5
Chloroethane		2000	300	100	50	20	10	4	1	0.5
Chloromethane		2000	300	100	50	20	10	4	1	0.5
Dichlorodifluoromethane		2000	300	100	50	20	10	4	1	0.5
Trichlorofluoromethane		2000	300	100	50	20	10	4	1	0.5
Vinyl Chloride		2000	300	100	50	20	10	4	1	0.5
Cyclohexanone	CYC	6250	3750	1250	625	500	250	200	50	25
2-Chloroethyl Vinyl Ether	2CEVE	5000	300	100	50	20	10	4	1	0.5
1,3-Butadiene	1,3-BUT	1000	300	100	50	20	10	4	1	0.5

Figure 1 Continued

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
EPA SW846 Method 8260A/B/C

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb	0.5 ppb
Acrolein	VACR	125000	3000	1000	500	200	100	40	10	5
tert-Amyl ethyl ether	TAEE	2000	300	100	50	20	10	4	1	0.5
Ethyl Ether	EE	1000	300	100	50	20	10	4	1	0.5
n-Pentane	n-PEN	1000	300	100	50	20	10	4	1	0.5
Freon 123a	Custom V	1000	300	100	50	20	10	4	1	0.5
Dichlorofluoromethane	LG Freon	1000	300	100	50	20	10	4	1	0.5
Hexachloroethane	VOA8	5000	300	100	50	20	10	4	1	0.5
2,2'-oxybis(1-Chloropropane)		5000	300	100	50	20	10	4	1	0.5
Ethanol	EOH	12500	7500	2500	1250	1000	500	250	62.5	31.25

ppb of analytical standard = (stock ppm)(µL stock) / flask mL

Analyst:_____
Date:_____

Appendix D – Field Forms

	PROJECT NUMBER	WELL NUMBER SHEET OF
	WELL PURGE AND SAMPLING FIELD SHEET	

	CASING DIAMETER	GAL/FT OF CASING	L/FT OF CASING
DEPTH TO WATER (FT): =	**1 IN.**	**0.0408**	**0.154**
WELL DEPTH (FT):	**2 IN.**	**0.1632**	**0.618**
WATER COLUMN (FT): =	**4 IN.**	**0.6528**	**0.618**
GAL/FT OF CASING x	**6 IN.**	**1.4688**	**5.560**
CASING VOLUME (GAL) =	**8 IN.**	**2.611**	**9.884**
NO. OF VOLUMES min.(3) x	**10 IN.**	**4.0797**	**15.444**
PURGE VOLUME (GAL) =	**12 IN.**	**5.8748**	**22.240**

METHOD OF PURGING (circle one)

PUMP: SUB. BLADDER. PERIST. **OTHER:**
TIME ON:
FLOW RATE (ml/min):
PUMP TIME (min):
VOL. PURGED (gals):

BAILER : TEFLON, SS ,OTHER:
BAILER VOL.. (gal)
REQUIRED PULLS:
VOL. PURGED (gals):
OTHER:

Date Time	Water Volume Discharged (gal)	Water Level (ft BTOC)	Turbidity (NTU)	Temperature (°C)	pH	Conductivity (mS/cm)	ORP mV	Dissolved Oxygen mg/L	Remarks (color, odor, sheen, sediment, etc.)

SEDIMENT SAMPLE COLLECTION FORM

Project Name:
Date(s):

Project #:		**Date:**	
Sample Location ID:		**Time:**	
Sample #:		**Weather:**	
Samplers:			

Sample Information:

Sample Depth:		**Sampling Device:**	
Water Depth:			
Distance from River Bank:			
River Flow Rate:			
Field Decon:	Yes No Dedicated	**Sample Type:**	Grab Composite

Munsell Color:

Sample Description:

Other physical characteristics of water body at sample location:
(Water color, turbidity, odor, presence of sheens, dead/stressed vegetation, etc.)

Sample Comments/Description:

TIDEWATER INC
ENGINEERS / SCIENTISTS / PROGRAM MANAGERS

LOCATION:
WELL / BORING ID:

Page ___ of ___

Project number:	**Driller Contractor:**	**Latitude:**	
Contract Number:	**Driller:**	**Longitude:**	
Date Logged:	**Drilling Equpment:**	**Surface Elevation:**	**ft amsl**
Total Depth:	**Drilling Method:**	**Top of Case Elevation:**	**ft amsl**
Geologist/Logger:	**Boring Diameter:**	**Backfill Method:**	
Reviewed by:	**Sampler Type:**	**Well Installed:**	**___Yes ___No**
PID/FID Equipment:	**Gamma Scan Equip.:**	**Type:**	

Depth (feet bgs)	Lithology	USCS	Sample Description	Sample Intervals	Sample ID /Time	Radiological Readings: Downhole (cpm)	Radiological Readings: Core (cpm)	Methane (ppm)	PID Reading (ppm)	Depth (feet bgs)	Well / Boring Completion	Comments

2.5
5.0
7.5
10
12.5
15
17.5
20

5.0
10
15

GROUNDWATER SAMPLE COLLECTION FIELD SHEET

GENERAL INFORMATION

SITE NAME _____ PROJECT NO. _____

SAMPLE NO. _____ WELL NO. _____

DATE/TIME COLLECTED _____ PERSONNEL _____

SAMPLE METHOD _____

SAMPLE MEDIA:

SAMPLE QC DUPLICATE: YES NO DUPLICATE SAMPLE NO. _____

MS/MSD REQUESTED YES NO MS/MSD SAMPLE NO.

SAMPLE CONTAINERS, PRESERVATIVES, ANALYSIS

Sample Container	Preservative	Analysis Requested

Volume per linear ft of casing		
ID (in)	Gallons	Liters
1	0.0408	0.1544
1 1/2	0.0918	0.3475
2	0.1632	0.6178
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600
8	2.611	9.8837

WELL PURGING DATA

Date _____ Well Depth (ft BTOC) _____

Time Started _____ Depth to Water (ft BTOC) _____

Time Completed _____ Water Column Length _____

PID Measurements Well Casing Volume (per ft) _____

Background _____ Volume of Water in Well (L) _____

Breathing Zone _____ Casing Volumes to Purge _____

Well Head _____ Minimum to Purge (L) _____

Purge Water _____ Actual Purge (L) _____

FIELD MEASUREMENTS

Time	Amount Purged (gal)	pH	Temperature (Celsius)	Conductivity (mS/cm)	Dissolved Oxygen (mg/L)	ORP (mV)	Turbidity (NTU)	Depth to Water (ft BTOC)	Purge Rate (mL/min)

FIELD EQUIPMENT AND CALIBRATION

Equipment	Model	Calibration

GENERAL COMMENTS _____

Multi-Parameter Probe Unit #

Field Parameters Measured in Flow-Through Cell

Pump Placement Depth =

Pump Rate =

Well Diameter =

Screen Interval =

Monitoring Well Construction Log Form

Project Name: _____ **Well No.** _____

Project Location: _____ **Project No.** _____

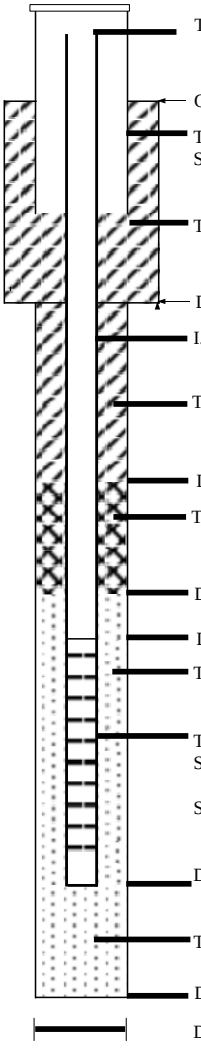
Installed By: _____ **Observed By:** _____ **Date of Well Completion** _____

Method of Installation: _____

Well Type (circle one): Single Cased Double Cased

Coordinates: Northing _____ **Survey Datums:** _____

Easting _____ _____



	feet bgs	Elevation (feet MSL)
Top of Casing		_____
Ground Elevation		_____
Type of surface casing _____ Surface Casing ID _____		
Type of surface seal _____		
Depth of surface seal	_____/	_____
I.D./Type of riser pipe _____		
Type of grout _____		
Depth to top of seal	_____/	_____
Type of seal _____		
Depth to top of filter pack	_____/	_____
Depth to top of screen	_____/	_____
Type of filter pack _____		
Type of screen _____ Screen ID _____		
Screen slot size _____		
Depth to bottom of well	_____/	_____
Type of backfill _____		
Depth to bottom of boring	_____/	_____
Diameter of boring _____		

Diagram Not To Scale

Notes:
bgs = Below ground surface
MSL = Mean sea level
NA = Not applicable
ID = Inside diameter

Focused Radiological Investigation Log Form

Project Name:		**Inv ID:**	
Project Location:		**Date:**	
Team Leader:		**Start Time:**	

Intrusive Investigation Coordinates:

Surface Data Collection:

Count Rate:

Dose Rate (Waist Level):

Dose Rate (At Ground Surface):

Surface Soil Sample ID:

Surface Soil Sample Collection Time:

Subsurface Data Collection:

Anomaly Depth:

Count Rate:

Dose Rate (Waist Level):

Dose Rate (At Ground Surface):

Subsurface Soil Sample ID:

Subsurface Soil Sample Collection Time:

Description of Radiological Item:

Size of Radiological Item:

Weight of Radiological Item:

Description of Surrounding Geologic Material:

Additional Notes/Observations:

Health and Safety Triggers:	**Description**
Methane detected at levels greater than 10% of the lower explosive limit	
VOCs detected by the PID in the breathing zone at levels that exceed 100 ppm	
Hydrogen sulfide in the work area at levels greater than 1 ppm	
Radiological dose rate readings observed at or above 0.5 mR/hr	