

SAMPLING AND ANALYSIS PLAN KERR-McGEE REFINING CORPORATION CUSHING, OKLAHOMA

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SAMPLING AND ANALYSIS PLAN KERR-McGEE REFINING CORPORATION CUSHING, OKLAHOMA

INTRODUCTION

Kerr-McGee Refining Corporation (KMRC), has prepared this Sampling and Analysis Plan for its refinery site in Cushing, Oklahoma. The Sampling and Analysis Plan describes in detail collection protocols for groundwater and surface and surface and subsurface soil samples. Also included are details on and and laboratory quality assurance/quality control programs, laboratory analyses, and monitor well installation and abandonment procedures.

This document is to be used by all facility personnel and consultants responsible for obtaining types of samples described above or who are responsible for monitor well installation or abandonment. A copy of this plan will be kept on file at the facility for appropriate personnel to review and reference.

GROUNDWATER SAMPLING PROTOCOL

Introduction

Groundwater sampling from wells is required to detect, define, and document any potential impacts to native groundwater quality resulting from facility operations. In order to assure that groundwater quality data is both accurate and representative of existing groundwater quality, the sampling personnel must be certain that the sample collected is representative of the groundwater that exists in the vicinity of the monitoring well, and that the chemical composition of the extracted groundwater has not been affected by the sampling methods.

Sampling Team Members

It is the responsibility of Kerr-McGee Corporation's Hydrologic Field Assistants to collect all routine groundwater monitor well samples at the facility. At least one of them is present during sample collection, although KMRC will supply an assistant to help purge monitor wells and to assist in the sampling.

STEPS TAKEN PRIOR TO SAMPLE WITHDRAWAL

Initial Observations

Field personnel who sample KMRC groundwater monitor wells note on an appropriate field parameter form (Table 1) the general well and site conditions upon arrival. For example, general comments about well damage or weather conditions for each sampling date are recorded. Specific comments on each of the wells are then noted, with typical notations including but not limited to, standing water around the well, the presence of nearby desiccation cracks, etc. A notation concerning missing well locks or caps is made, if appropriate. Observations related to the physical condition of the well are recorded if it appears the well has been damaged (as a result of vandalism, traffic, etc). It is the responsibility of the hydrologic field assistant to submit a written request for maintenance to the facility site coordinator.

Measurement of Static Water Level and Well Depth - After all general and pertinent observations are recorded, the hydrologic field assistant will measure a static water level. The well cap is removed and laid upside down on the ground or placed in the field vehicle. An electric water level indicator or oil/water interface probe is tested to make sure it is operable. The detection probe is then washed with a non-phosphate detergent and rinsed with deionized water. The probe is lowered downhole until the fluid level is detected. If a floating immiscible liquid is encountered, the probe is then lowered until it contacts groundwater. The depth to both fluids is then recorded (on the field parameter form) to the nearest 0.01 foot with respect to a reference point (either a mark or a notch) at the top of the

casing. All reference points have been measured in relation to mean sea level by a licensed surveyor.

Total well depth is then measured with respect to the top of the casing for each well using the water level indicator or interface probe. Again, the measurement is recorded (on the field parameter form) to the nearest 0.01 foot. The measuring device is removed from the well, washed with a non-phosphate detergent, rinsed with deionized water, and dried with a disposable paper towel.

Measurement of Immiscible Fluids - The presence of a floating or settled immiscible liquid in a well is recorded on the field parameter form. Floating liquids are often detected visually by gently lowering a dedicated clear sampling tube (bailer) to just below the air/fluid interface. The bailer is retrieved from the well and the thickness of the "floater", if a floating immiscible layer is present, is measured using a steel tape marked with 0.01 foot graduations. When an oil/water interface probe is used instead of the clear bailer, the depth from the top of the casing to each fluid is read directly and recorded on the field parameter form.

If the presence of immiscible "sinkers" is believed to be present in site monitor wells, then the interface probe will be used to measure their thickness. There is, however, no reason to believe immiscible "sinkers" are present at the site.

Withdrawing a Sample of Immiscible Fluid - Prior to sampling of immiscible fluid in a well, the thickness of the fluid is measured

as detailed above. If a sufficient thickness of fluid is present (generally greater than one foot), a standard, bottom- filling bailer will be used to withdraw the sample. The bailer will be slowly lowered with care to prevent allowing water to be withdrawn with the fluid. A fluid sample can then be poured from the top of the bailer. The bailer will then be discarded.

If the immiscible fluid is not sufficiently thick, a top-filling bailer with a stopcock in the bottom will be lowered until the top is near the fluid/water interface. The bailer will be removed and the stopcock opened to allow water that has accumulated to drain. The fluid sample will then be poured into sample containers through the bottom of the bailer.

Withdrawing a Water Sample

<u>Calculating Volume of Water to be Evacuated</u>- As a standard practice, a minimum of three casing volumes of water will be removed from each well before obtaining a sample. There are instances, however, where well recovery is extremely slow and three

casing volumes cannot be removed. When this occurs, the well will be pumped or bailed to near dryness. The well will then be sampled after a sufficient volume of water is available to obtain enough sample for the required analyses. The volume of water purged from each well prior to obtaining a sample is recorded (on the field parameter form) by the field person.

KMRC has prepared a table (Table 2) used by the hydrologic field assistant in determining the amount of fluid to be evacuated from

various size monitor wells. If the circumstances occur where the table is no applicable, the field person will calculate the volume of fluid to be removed by using the conversion factors of 0.16, 0.37, 0.65, 1.02 and 1.47 gallons per foot of water in well for 2, 3, 4, 5 and 6 inch diameter wells, respectively. For example, given a 2 inch well with 40 feet of standing fluid:

40 ft. x 0.16 gal/ft = 6.4 gal. per casing volume therefore, 19.2 gallons of fluid (3 casing volumes) would need to be evacuated before a sample is taken.

Well Evacuation- Well evacuation for stainless steel monitor wells is performed with the use of peristaltic pumps or dedicated stainless steel bailers and attached Teflon coated wire. In the case of PVC monitor wells, evacuation is done with dedicated PVC bailers or peristaltic pump. When not in use, the bailer and rope are stored inside of the well and are protected from contact with the groundwater. When a peristaltic pump is used for well excavation, that portion of the tubing (the suction line) that extends into the groundwater will be cut off and disposed of before using the pump to evacuate the next well.

Just prior to well evacuation with a bailer, a clean plastic sheet or a 30 gallon trash can lined with clean plastic is placed adjacent to the monitor well to prevent the bailer and rope from contacting the ground. This procedure is done to prevent the bailer and rope from possibly being contaminated from touching the ground during the bailing and sampling process and therefore affecting the groundwater analyses.

When well evacuation is performed with a bailer, the bailer is gently lowered downhole to prevent de-gassing of the fluid column. The bailer is allowed to fill and is then retrieved from the well. All purged water (from either a bailer or by pumping) is poured into and retained in a 55 gallon drum placed adjacent to the well. After groundwater sampling has been performed, the 55 gallon drum is emptied into one of the site waste pits. The sampler will note on the field parameter form observations concerning the purged water (i.e., odor, color, oils, etc.).

When well purging is done with a pump, the pumping rate is kept at 3 gpm or less to reduce the possibility of loss of volatiles. Polyethylene or PVC tubing will be used to evacuate the well. The end of the suction line will extend approximately one foot below the water level to ensure that water entering the screen flows up and no "stagnant" water is left in the well casing.

In the case of poorly yielding wells, where three casing evacuations cannot be withdrawn, the well is bailed or pumped to dryness, and allowed to recover until enough sample volume can be removed for the necessary analyses. This may require returning to a well several hours after it was initially evacuated or on the following day to obtain an adequate groundwater sample. Whereas well evacuation/purging is done by bailer or peristaltic pump, all sampling is done with the use of dedicated bailers.

Field Measurements and Field OA/OC Procedures- After the necessary casing evacuations, a bail or two of water is divided into 8 clean polypropylene beakers. Four of the beakers will provide

quadruplicate analyses of pH and the others will provide quadruplicate field determinations of temperature and specific conductivity. Before the four replicate pH measurements are performed, the pH meter is calibrated following manufacturer recommendations in pH 7 and pH 4 buffer solutions. Following calibration, the pH probe is rinsed in distilled or deionized water and immersed in each of the four beakers for pH determination. The pH of the sample is allowed to equilibrate in each beaker before the reading is recorded on the field parameter form. The samples are then discarded into the 55 gallon collection drum and the beakers are rinsed with deionized water.

Four replicate conductivity measurements are made in each of the remaining four beakers following calibration of the conductivity meter. The meter is calibrated according to manufacturer recommendations, followed by a check of the probe in 0.01 N KCl solution. The conductivity of the standard solution should be approximately 1,413 umhos/cm. The conductivity probe is then rinsed with deionized water and placed in each of the field samples. Allowing 2-3 minutes for equilibration, the conductivity and temperature of each of the four samples is then recorded (field parameter form), followed by a thorough rinsing of the probe in deionized water. The samples are then discarded into the 55 gallon collection drum and the beakers are rinsed with deionized water.

A field logbook will be maintained at the facility. The field logbook will contain the following information for each sampling event:

* date and time of purging and sample collection,

- * well number, depth to water, casing size,
 - * volume of water purged and equipment used,
 - * field data (including pH and specific conductivity)
 - * sample identification numbers,
 - * parameter for analysis, containers, and preservatives used,
 - * field observations,
 - * sample destination and transportation method,
 - * sampling personnel, and
 - * deviations from the sampling and analysis plan

Copies of the field parameter form and chain of custody form may be included in the field logbook. Because of the use of dedicated sampling devices (bailers) field blanks are not collected. Trip blanks are provided by the laboratory only when sampling events include analyses for volatile organic compounds.

When trip blanks are required, one trip blank consisting of two 40-ml VOA bottles, filled with deionized water at the laboratory, will be included for each day's sampling. Trip blanks will be analyzed for the same volatile constituents and by the same method as the water samples they accompany.

Sample Collection- As the pH and conductivity determinations are being made, the hydrologic field assistant collects the necessary volume of sample from each well with a bailer for the laboratory analyses. Water samples for laboratory analyses are poured from the bailer into appropriate clean containers. Sample containers for organic analyses are amber glass bottles with teflon-lined caps. Samples requiring volatile organic analyses are small glass vials with teflon septa. Samples for inorganic metal analyses are collected in either glass or plastic containers. All sample containers are filled to minimize headspace and reduce the potential for change in certain volatile constituents. Table 3

TABLE 3 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES WATER SAMPLES CUSHING, OKLAHOMA SAMPLING AND ANALYSIS PLAN

TEST FOR	CONTAINER	PRESERVATION	HOLDING TIM				
Total Organic Carbon (TOC)	(1) 500 ml amber glass bottle	Cool to 4°C HCl or H ₂ SO ₄ to pH <2	28 days				
Total Organic Halogen (TOX)	(1) 500 ml amber glass bottle	Cool to 4° C H ₂ SO ₄ to pH <2	7 days				
Specific Conductivity	(1) 500 ml plastic bottle	Cool to 4°C	28 days				
рН	(1) 500 ml plastic bottle	Cool to 4°C	72 hours				
Total Petroleum Hydrocarbon (TPH) by GC/FID	(1) 1 liter glass bottle	Cool to 4°C	28 days				
Total Petroleum Hydrocarbon (TPH) by IR	(1) 1 liter glass bottle	Cool to 4°C	28 days				
TCLP Metals (leachable metals)	(1) 1 liter glass bottle	Cool to 4°C	6 months				
Skinner List Metals (total metals)	(1) 1 liter plastic bottle	Cool to 4°C HNO3to pH <2	6 months				
Benzene, Toluene, Ethylbenzene, Xylene (BTEX)	(2) 40 ml VOA vials	Cool to 4°C	7 days				
TCLP Volatiles	(2) 40 ml VOA vials	Cool to 4°C	14 days				
Skinner List Volatiles	(2) 40 ml VOA vials	Cool to 4°C	7 days				
TCLP Semi- Volatiles	(1) 1 liter glass amber	Cool to 4°C	7 days				
Skinner List Semi-Volatiles	(1) 1 liter glass amber	Cool to 4°C	7 days				





lists the collection container type and size for each analysis performed. Sample containers are filled in the following sequence:

1.	Volatile organics	8.	Dissolved metals
2.	Purgeable organic carbon	9.	Phenols
3.	Purgeable organic halogens	10.	Cyanide
4.	Total organic halogens	11.	Sulfate/chloride
5.	Total organic carbon	12.	Turbidity
6.	Extractable metals	13.	Nitrate/ammonia
7.	Total metals	14.	Radionuclides

Sample Preservation and Shipment- Immediately after collection, all samples are stored in an insulated sample shuttle (chest) and cooled to 4°C with either "blue ice" or a plastic bag filled with ice. Those samples to be analyzed for total organic carbon are additionally preserved with 1-2 ml of ultrapure hydrochloric or sulfuric acid, while those samples designated for total organic halogen analysis are preserved with ultrapure sulfuric acid.

Total metals samples are preserved with nitric acid. Filtration and further preservation procedures (as required) are performed at the laboratory following receipt of the samples.

Samples are shipped in the above-mentioned insulated shuttle on the same day they are collected. All samples are shipped via overnight express delivery service to the laboratory. All groundwater samples are accompanied by chain-of-custody forms. A sample chain of custody form is included as Table 4.

Field Chain of Custody Forms



forms, and are sealed. Upon arrival at the laboratory, the shuttle is inspected and opened and the chain of custody form is signed with the date and time opening occurred. Additional sample preservation and storage information are logged on a laboratory chain of custody form from this point forth.

Laboratory Sample Custody Log

Once the sample shuttle arrives at the laboratory, the shuttle seal is broken by the designated sample custodian. Each sample is checked and stored in a secure area prior to analysis. The sample custodian records the disposition of each sample to an analyst or technician on a sample custody log. The use of the sample in each sample bottle is also documented along with the appropriate testing procedure. The record shows for each link in the process the person with custody and the date each person accepted or relinquished custody.

Split Sampling Events

In the event an authorized regulatory agency wishes to obtain split samples, it is the agency's responsibility to supply their own collection containers and preservatives. Well purging will be done according to this Sampling and Analysis Plan, and sample containers will be filled alternately between KMRC and the regulatory agency.

Introduction

The potential impact(s) to surface water quality from groundwater discharges or facility operations must be defined and documented. All sampling procedures listed below must be followed to insure the collection and analysis of representative surface water samples.

Steps Taken Prior to Obtaining Water Samples

Field personnel who obtain stream samples for KMRC record site weather conditions for the sampling date on the field parameter form. A notation is made for unusual weather events just prior to sampling (for example, drought, heavy rain previous week, etc). A stream elevation measurement is taken relative to a stationary benchmark installed on the bank near each stream sampling location and is recorded to the nearest 0.01 foot. The presence of a floating or settled immiscible liquid in the stream is made. Other notations are made if deemed appropriate.

Obtaining a Water Sample

Sample Collection- Grab samples of the stream are taken either from the shore or by wading into the stream, turning toward the upstream direction, and dipping the sample bottles to just below the water surface. Filling and capping the bottles under water eliminates the potential of headspace in the bottles; this is particularly important for the measurement of volatile organics. Bottles that have already been prepared with preservatives by the lab (for example, TOC bottles with sulfuric acid) are not dipped under water, but rather are filled with fluid poured from one of the other bottles. This eliminates the loss of preservative that would occur if these bottles were filled under water. Any sample transfer between bottles is performed carefully and slowly, avoiding turbulence, which could result in the loss of volatiles.

Field Measurements and Field QA/QC Procedures- Identical field measurement and field QA/QC procedures as described for groundwater samples (see p. 10) pertain to stream samples. Refer to that section for complete details.

<u>Sample Preservation and Shipment</u>- The sample preservation and shipment procedures described in the groundwater sampling section (see p. 14) pertain to samples collected fr m streams. Refer to that section for complete details.

Field Chain of Custody

The field chain of custody protocol described in the groundwater sampling section (see p. 14) also pertains to surface water sampling. Refer to that section for complete details.

Laboratory Sample Custody Log

Laboratory sample custody for stream samples is identical to that described in the groundwater sampling section (see p. 16). Refer to that section for complete details.



SURFACE AND NEAR SURFACE SOIL SAMPLING PROTOCOL

Introduction

Surface and near surface soil samples (depth: 0 to 1 meter) will be collected following procedures described in "Preparation of Soil Sampling Protocol: Techniques and Strategies", an EPA document prepared by Benjamin J. Mason (May 1983). A copy of the appropriate section of this document is contained in Appendix A. Generally, the procedures allow for the use of soil punches, scoops and shovels, soil probes and augers, and power augers.

Initial Observations

Soil sampling locations will be marked in the field and referenced on field parameter and chain of custody forms so as to enable re-sampling of that exact location at a later date, if necessary. Weather conditions on the sampling date will be described as will any unusual weather events (for example, drought) just prior to the sampling. Other appropriate notes will be made as deemed necessary.

Obtaining Soil Samples

Sampling Tools- Depending upon the required laboratory analyses, soil samples will be collected using various methods. Surface and near surface samples requiring chemical analyses (for example, wet chemistry methods) will be collected with soil punches, scoops, shovels, probes, or augers and stored in glass jars. Those samples to be tested for physical parameters (for example, permeability, Atterberg Limits, etc.) will be collected using dedicated Shelby (thin walled) tubes advanced by a drill rig, if possible. Both

ends of the Shelby tube will be capped and secured upon retrieval from the hole to reduce disturbance of the sample. If the use of a thin walled tube is not possible, a sample will be obtained with a split-barrel sampler and will be placed in a glass jar.

Soil samples collected with scoops, shovels, probes and augers will be described according to ASTM Method D2488, Description and Identification of Soils (Visual-Manual Procedures).

Field Compositing- Where composite samples must be taken, compositing will be done with large dedicated plastic sheets (one time use only) or with stainless steel mixing bowls. In each case, clods of soil will be broken up before being mixed with hand tools. Following mixing, aliquots will be placed in piles and a small sample from each aliquot will be taken and mixed together to form the composite. The composite will then be placed in a glass jar and shipped with the rest of the samples to the laboratory. The excess soil will be discarded. Table 5 presents the sample containers and holding times for soil analyses.

<u>Sample Preservation</u>- Soil samples collected in jars for laboratory analyses will be stored at 4°C in the sample shuttle pack and transported to the laboratory via overnight express delivery. When Shelby tube samples are retained for physical soil testing, plastic

TABLE 5 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES SOIL SAMPLES CUSHING, OKLAHOMA SAMPLING AND ANALYSIS PLAN

TEST FOR	CONTAINER	PRESERVATION	HOLDING TIME
Total Organic Carbon (TOC)	(1) 500 ml wide mouth jar	Cool to 4°C	28 days
Total Organic Halogen (TOX)	(1) 500 ml wide mouth jar	Cool to 4°C	14 days
рH	(1) 250 ml wide mouth jar	Cool to 4°C	72 hours
Total Petroleum Hydrocarbon (TPH) by GC/FID	(1) 250 ml wide mouth jar	Cool to 4°C	28 days
Total Petroleum Hydrocarbon (TPH) by IR	(1) 250 ml wide mouth jar	Cool to 4°C	28 days
TCLP Metals (leachable metals)	(1) 500 ml wide mouth jar	Cool to 4°C	6 months
Skinner List Metals (total metals)	(1) 250 ml wide mouth jar	Cool to 4°C	6 months
Benzene, Toluene, Ethylbenzene. Xylene (BTEY)	(1) 125 ml VOA vial	Cool to 4°C	14 days
TCLP Volatiles	(1) 125 ml	Cool to 4°C	14 days
Skinner List Volatiles	(1) 125 ml VOA vial	Cool to 4°C	14 days
TCLP Semi- Volatiles	(1) 500 ml wide mouth jar	Cool to 4°C	14 days
Skinner List Semi-Volatiles	(1) 500 ml wide mouth jar	Cool to 4°C	14 days

caps will be taped on both ends of the shelby tube. This will retain moisture so that physical characteristics will be maintained. No other special preservation procedures will be utilized for soil samples collected by this method.

Decontamination Procedures

All sampling implements will be decontaminated between use with a steam cleaner or in a non-phosphate detergent solution (i.e., Alconox or similar), followed by rinsing with clean water.

Field Chain of Custody

Chain of custody forms will accompany all soil samples collected and shipped for analyses. A sample chain of custody form is included as Table 4 (See p. 15).

Laboratory Sample Custody Log

A laboratory sample custody log will accompany all samples undergoing analyses. A complete description of this procedure is described in the groundwater sampling section (see p. 16).



Introduction

Subsurface soil sampling refers to those procedures used to obtain soil samples from a depth of greater than 1 meter. In some soil samples from a depth of greater than 1 meter. In some instances, small portable power augers may be useful to slightly greater depths, but generally the procedures require the use of a drill rig and Shelby tube (thin wall) or split-barrel (split-spoon or continuous sampler) devices.

Initial Observations

Borehole locations will be referenced on field parameter and chain of custody forms to permanent/semi-permanent structures and located on facility site drawings. (In some cases, it may be necessary to survey exact locations of the borings). Weather conditions on the sampling date will be described as well as any unusual weather conditions occurring prior to the sampling date. Other appropriate notes may be made as deemed necessary.

Obtaining Sub-Surface Soil Samples

<u>Sample Collection</u>- Some borings will require either continuous or intermittent sampling from grade level to total depth. Such samples will be collected by either the use of dedicated Shelby (thin walled) tubes or split-barrel (split-spoon or continuous sampler) devices. Shelby tube samples will be collected following the method described in ASTM Method D1587 and split-spoon samples will be collected following ASTM Method D1586. All boreholes will be visually logged from grade level to the target depth. Soil and

formation description will follow ASTM Method D2488, along with notes concerning water level and visual or olfactory evidence of contamination. A photo-ionization detector or comparable instrument will be used in the field to screen logged samples for volatile constituents.

Borings not completed as monitor wells will be grouted back to the surface using a tremie line and grout consisting of Type I Portland cement with at least 5% bentonite.

Field Compositing- Where composite samples must be taken, compositing will be done with large dedicated plastic sheets (one time use only) or with stainless steel mixing bowls. In each case, clods of soil will be broken up before being mixed with hand tools. Following mixing, aliquots will be placed in piles and a small sample from each aliquot will be taken and mixed together to form the composite. The composite will then be placed in a glass jar and shipped with the rest of the samples to the laboratory. The excess soil will be discarded.

<u>Sample Preservation</u>- Soil samples collected off auger flights or from the split barrel for wet chemistry analyses will be stored in glass jars and kept at 4°C until delivered to the laboratory via overnight express delivery. No special preservation will be 24 performed for Shelby tube samples other than capping both ends of the tube after removal from the borehole.

Decontamination Procedures All sampling implements will be decontaminated between use with a steam cleaner or in a detergent

solution (i.e. Alconox or similar), followed by rinsing with clean water.

Field Chain of Custody Chain of custody forms will accompany all soil samples collected and shipped for analysis. A sample chain of custody form is included as Table 4 (see p. 15).

Laboratory Sample Custody Log A laboratory sample custody log will document sample disposition in the laboratory (see p. 16 for complete details).

ANALYSIS OF ALL WATER AND SOIL SAMPLES

Wet Chemical Analyses

All water and soil samples submitted to the contract analytical laboratory for analyses will be handled and analyzed in accordance with the highest standards. Most analytical methodologies originate from USEPA SW-846; several methods are referenced to EPA procedures and one to Standard Methods. Tables 6 and 7 have been prepared to summarize the methods the contract laboratory will use for both the water and soil samples.

Detection limits for inorganic compounds are listed in the laboratory QA/QC program, included as Appendix B. Detection limits for organic compounds, where relevant, are those stipulated by the methods and protocols listed in SW-846. Presentation of detection limits of all compounds is not possible, since dilution factors stipulated in SW-846 will cause variation in those limits.

Physical (Soil) Testing

Some soil samples may be submitted to a soils-testing laboratory for the determination of physical properties of that sample. Soil testing parameters have not been specified, but may include such parameters as 1) particle size analysis (ASTM Method D421/D422); 2) permeability- constant head (ASTM Method D2434); 3) Atterberg Limits (ASTM Method D4318). Other required physical testing will also comply with ASTM specifications, when applicable. TABLE 6 ANALYTICAL METHODOLOGIES FOR WATER SAMPLES CUSHING, OKLAHOMA SAMPLING AND ANALYSIS PLAN

PARAMETER	SW 8 PREPARAT	46 ION METHOD	EPA METHOD	STANDARI METHOD
Conductivity pH TOC TOX		9050 9040 9060 9020		
Chloride Iron Manganese Sodium Sulfate Barium Cadmium Fluoride Nitrate Mercury Selenium Silver Arsenic Chromium Lead Cyanides	3010 3010 3010 3020 3020 3020 3010 3010	6010 6010 6010 6018 7470 7740 6010 7060 6010 6010 9010	325.2 375.3 353.2	413E
Total Recoverabl Petroleum Hydro	le ocarbons			418.1
Acid/Base/Neutra Extractables	al		3540/8270	þ
Volatile Organic	CS		5020/8240	0





TABLE 7 ANALYTICAL METHODOLOGIES FOR SOIL SAMPLES CUSHING, OKLAHOMA SAMPLING AND ANALYSIS PLAN

PARAMETER	SW 846 PREPARATION	METHOD	EPA <u>METHOD</u>	STANDARD METHODS
Conductivity pH TOC TOX		9050 9040 9060 9020		
Chloride	1.1.1 - Fill (1996)		325.3	
Iron	3050	6010		
Manganese	3050	6010		
Sodium	3050	6010		
Sullate	2050	6010	375.3	
Gadmium	3050	6010		
Fluoride	3030	0010	240 2	
Nitrate			352 1	
Mercury		7471	556.1	
Selenium	3020	7740		
Silver	3050	6010		
Arsenic	3020	7060		
Chromium	3050	6010		
Lead	3050	6010		
Cyanides				412D
Total Recoverab Petroleum Hydro	le carbons		418.1	
Acid/Base/Neutr Extractables	al	3540/82	70	
Volatile Organi	CS	5020/824	40	
TCLP	1311*			

*40 CFR Part 261, Appendix II



Laboratory OA/OC Program

The contract analytical laboratory currently performing the wet chemical analyses has provided a copy of their QA/QC program. This information is provided in Appendix B. Because physical testing of the soils has not yet been defined, a laboratory QA/QC program for these tests has not been prepared. All testing, however, will be done in accordance with ASTM specifications. All groundwater monitor wells will be installed so as to yield representative groundwater quality data. Installation methods will include the use of hollow stem auger or rotary wash drilling techniques with minimal introduction of drilling fluid into the borehole.

Temporary wells may remain open-hole completed if all that is required is a water level elevation measurement or a grab sample for chemical analysis. A temporary well may be completed by placing a PVC screen at the desired level. Temporary wells will be plugged either by cementing the hole with a tremie line from total depth to grade using a neat Portland Type I cement with 5% bentonite or by returning the cuttings to the borehole. Monitor wells will be abandoned by cutting the casing at or just below grade level and using a tremie line to pump a cement/bentonite grout from total depth to grade. Well abandonment/plugging data will be reported to the appropriate regulatory agencies.

Wells installed for long term monitoring will be 2 inches or larger in diameter and constructed of PVC, stainless steel or teflon material, depending upon agreement with the regulatory agency. All joints will be flush threaded without the use of cementing compounds. If PVC pipe is used, it will bear the National Sanitation Foundation logo for potable water applications.

Monitor well completion will be in accordance with the guidelines contained in the "RCRA Groundwater Monitoring Technical Enforcement

Guidance Document" (1986). Specifically, the annular space between the screen and borehole will be filled with a filter pack of proper gradation to provide mechanical retention of the formation sand and silt. The filter pack will extend to between one and two feet above the top of the well screen. A minimum of two feet of bentonite pellets will be placed immediately above the filter pack in the annular space between the well casing and borehole.

Above the bentonite seal will be a cement/bentonite grout mixture consisting of 3 to 5 pounds of bentonite per 94 pound sack of cement with approximately 6.5 gallons of water. A tremie line will be used to place the grout from the top of the bentonite seal to three feet below grade level. Following a suitable amount of time to allow for grout settlement, the annular space from three feet below grade to grade level will be sealed with concrete, blending into a cement apron extending three feet from the outer edge of the borehole. Figure 1 illustrates a permanent well completion, as per the "Groundwater Monitoring Technical Enforcement Guidance Document" (1986).

A variation from this installation format may be necessary in some situations because of an extremely shallow water table. In such instances, the distance from the top of the screen to the top of the filter pack thickness may be reduced to as little as 0.5 feet, and the bentonite pellet seal thickness may be reduced to one foot. No grout will be poured in the annulus, and the concrete pad will be poured on top of the bentonite pellet seal. Such variations are only necessary for monitor wells that must intersect the water table, when the water table is less than six feet below grade.

Following well completion, well development will be performed to remove any fluids used during drilling and to remove fines from the natural formation. This will provide a particulate free discharge for sampling. Development will be done by reversing flow direction or surging the well. If possible, no fluids other than natural formation water will be added during development. A locking cap will be placed on all wells.

A completed soil boring log (Figure 2) form for each soil boring performed and monitor well installation diagram form (Figure 3) for each monitor well installed will be retained on site. These records will include:

- date/time of construction
- drilling method/fluid used
- well location (within 0.5 ft.)
- borehole diameter and well casing diameter
- well depth (within 0.1 ft.)
- drilling and lithologic logs
- depth to first saturated zone
- casing material
- screen material and design
- casing and screen joint type
- screen slot size/length
- filter pack material/size
- sealant materials
- sealant placement method
- surface seal design/construction
- well development procedure
- type of protective well cap

- ground surface elevation
- top of casing elevation (to 0.01 ft MSL)
- detailed drawing of well (including dimensions)

All wells will be permanently labeled. Well locations and top-ofcasing elevations will be surveyed by a licensed surveyor. Locations will be surveyed to within 1.0 ft. and the elevation of the top of each well casing will be measured to within 0.01 ft. above mean sea level elevation. Well locations will be plotted on the facility base map. REFERENCES

- Annual Book of ASTM Standards Section 4, Volume 04.08, 1990, ASTM, Philadelphia, PA, 1092 p.
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- Standard Methods for the Examination of Water and Wastewater-15th Edition, 1980, AOHA-AWWA-WPCF, Washington, D.C.
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APPENDIX A

SOIL SAMPLING PROTOCOL

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FB83-206979

PREPARATION OF SOIL SAMPLING PROTOCOL: TECHNIQUES AND STRATEGIES

by

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under subcontract to

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Cooperative Agreement Number: CR808529-01-2

Project Office

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7.1 Surface Sampling

Surface soil sampling can be divided into two categories -the upper 15 cm and the upper meter. The very shallow pollution such as that found downwind from a new source or at sites of recent spills of relatively insoluble chemicals can be sampled by use of one of the methods listed in Section 7.1.1. The deeper pollutants found in the top meter are the more soluble, recent pollutants or those that were deposited on the surface a number of years ago. These have begun to move downward into the deeper soil layers. One of the methods in 7.2 should be used in those cases.

7.1.1 Sampling with a Soil Punch

A number of studies of surface soils have made use of a punch or thin walled steel tube that is 15 to 20 cm long to extract short cores from the soil. The tube is driven into the soil with a wooden mallet; the core and the tube are extracted; the soil is pushed out of the tube into a stainless steel mixing bowl and composited with other cores. Two alternates are the short Kingtube samplers or the tube type density samplers used by the Corps of Engineers. (These sampling devices can be supplied by any field equipment company or by agricultural equipment companies.) The latter sampler is machined to a predetermined volume and is designed to be handled and shipped as a soil-tube unit. A number of similar devices are available for collecting short cores from surface soils.

The soil punch is fast and can be adapted to a number of analytical schemes provided precautions are taken to avoid contamination during shipping and in the laboratory. An example of how this method can be adapted would be to use the system to collect samples for volatile organic chemical analysis. The tubes could be sealed with a Teflon plug and coated with a vapor sealant such as paraffin or, better yet, some non-reactive sealant. These tubes could then be decontaminated on the outside and shipped to the laboratory for analyses.

7.1.2 Ring Sampler

Soil engineers have a tool that can be purchased from any engineering equipment supply house that can be used to collect larger surface samples. A seamless steel ring, approximately 15 to 30 cm in diameter, is driven into the soil to a depth of 15 to 20 cm. The ring is extracted as a soil-ring unit and the soil removed for analysis. These large cores should be used where the results are going to be expressed on a per unit area basis. This allows a constant area of soil to be collected each time. Removal of these cores is often difficult in very loose sandy soil and in very tight clayey soils. The loose soil will not stay in the ring. The clayey soil is often difficult to break loose from the underlying soil layers thus the ring must be removed with a shovel.

This device has not been used extensively for collecting samples for chemical analysis but the technique should offer a

useful method for collecting samples either for area contamination measurements or for taking large volume samples.

7.1.3 Scoop or Shovel Sampling

Perhaps the most undesirable sample collection device is the shovel or scoop. This technique is often used in agriculture but where samples are being taken for chemical pollutants, the inconsistencies are too great. Samples can be collected using a shovel or trowel if area and/or volume are not critical. Usually the shovel is used to mark out a boundary of soil to be sampled. The soil scientist attempts to take a constant depth of soil but the reproducibility of sample sizes is poor; thus the variation is often considerably greater than with one of the methods listed above.

7.2 Shallow Subsurface Sampling

Precipitation may move surface pollutants into the lower soil horizons or move them away from the point of deposition by surface runoff. Sampling pollutants that have moved into the lower soil horizons requires the use of a device that will extract a longer core than can be obtained with the short probes or punches. Three basic methods are used for sampling these deeper soils.

- · Soil probes or soil augers
- · Power driven cores
- · Trenching

The soil probe collects 30 or 45 cm of soil in intact, relatively undisturbed soil cores whereas the auger collects a "disturbed" sample in approximately the same increments as the probe. Power augers can use split spoon samplers to extract cores up to 60 cm long. With special attachments longer cores can be obtained with the power auger if this is necessary.

The requirement for detail often desired in research studies or in cases where the movement of the pollutants is suspected to be through very narrow layers cannot be met effectively with the augers. In these cases some form of core sampling or trenching should be used.

7.2.1 Soil Probes and Hand Augers

Two standard tools used in soil sampling are the soil probe (often called a King-tube) and the soil auger. These tools are designed to acquire samples from the upper two meters of the soil profiles. The soil probe is nothing more than a stainless steel or brass tube that is sharpened on one end and fitted with a long, T shaped handle. These tubes are usually approximately 2.5 cm inside diameter although larger tubes can be obtained. The cores collected by the tube sampler or soil probe are considered to be "undisturbed" samples although in reality this is probably not the case. The tube is pushed into the soil in approximately 20 to 30 cm increments. The soil core is then removed from the probe and





placed in either the sample container or in a mixing bowl for compositing.

The auger is approximately 3 cm in diameter and is used to take samples when the soil probe will not work. The samples are "disturbed"; therefore, this method should not be used when it is necessary to have a core to examine or when very fine detail is of interest to the scientist. The auger is twisted or screwed into the soil then extracted. Because of the length of the auger and the force required to pull the soil free, only about 20 to 30 cm maximum length can be extracted at one time. In very tight clays it may be necessary to limit the length of each pull to about 10 cm. Consecutive samples are taken from the same hole thus cross contamination is a real possibility. The soil is compacted into the threads of the auger and must be extracted with a stainless steel spatula.

Larger diameter augers such as the bucket auger, the Fenn auger and the blade augers can also be used if larger samples are needed. These range in size from 8 to 20 cm in diameter.

If distribution of pollutant with depth is of interest, the augers and the probes are not recommended because they tend to contaminate the lower samples with material from the surface. The probe is difficult to decontaminate without long bore brushes and some kind of washing facility. One alternative is to take several waste cores at each site prior to collecting the actual samples. This allows the probe tube to be cleaned by the scouring action of soil at similar concentrations to those found in the sample taken. This should remove any contamination leftover from previous locations. Where there is a potential for litigation, decontamination is essential to avoid any question about cross contamination. The augers have some of the same decontamination problems but the open thread surfaces allow easier access to the collection surfaces; therefore, they are easier to clean. See Section 7.8 for more detail on decontamination Procedures.

One final warning about the use of the hand augers and soil probes. There are many soil scientists with back problems that have resulted from trying to extract a tool that has been inserted too far into the soil. A foot jack is a necessary accessory if these tools are to be used. The foot jack allows the tube to be removed from the soil without use of the back muscles.

7.2.2 Power Augers and Core Samplers

These truck or tripod mounted tools are used for collecting samples to depths greater than approximately 30 cm. Standard ASTM methods for use of these tools are these tools are available from the American Society for Testing and Materials or can be found at any college or university library. The methods outlined in Section 7.3 are applicable in this case and will not be discussed further.

7.2.3 Trenching

This method of soil sampling is used to carefully remove sections of soil during studies where a detailed examination of pollutant migration patterns and detailed soil structure are required. It is perhaps the least cost effective sampling method because of the relatively high cost of excavating the trench from which the samples are collected. It should therefore be used only in those cases where detailed information is desired.

A trench approximately 1 meter wide is dug to a depth approximately one foot below the desired sampling depth. The maximum effective depth for this method is about 2 meters unless done in some stepwise fashion. Where a number of trenches are to be dug, a backhoe can greatly facilitate sampling. The samples are taken from the sides of the pit using the soil punch or a trowel.

The sampler takes the surface 15 cm sample using the soil punch or by carefully excavating a 10 cm slice of soil that is 10 cm square on the surface. The soil can be treated as an individual sample or composited with other samples collected from each face of the pit. After this initial sample is taken the first layer is completely cut back exposing clean soil at the top of the second layer to be sampled. Care must be exercised to insure that the sampling area is clear of all material from the layers above. The punch or trowel is then used to take samples from the shelf created by the excavation from the side of the trench. This process is repeated until all samples are taken. The resulting hole appears as a set of steps cut into the side of the trench as is shown in Figure 7.1.

An alternate procedure that is also effective results from using the punch to remove soil cores from the side of the trench at each depth to be sampled (Figure 7.1). Care must be taken to guard against soil soughing down the side of the hole. A shovel should be used to carefully clean the soil sampling area prior to driving the punch into the trench side.

7.4 Compositing

Many sample plans call for compositing of the soils collected at a sampling location. This creates a problem from the point of view of the soil scientist. The key to any statistical sampling plan is the use of the variation within the sample set to test hypotheses about the population and to determine the precision or reliability of the data set. As was mentioned earlier, the composite sample provides an excellent estimate of the mean but does not give any information about the variation within the sampling area. Section 7.4.1 discusses one alternative that is a combination of the compositing methods and random sampling with duplication. Three methods that have been used to composite soil are presented below.

7.4.1 Estimating Sample Variance.

The problem with the statistical analysis 1 found in the lack of duplication within the sampling location. Each subsample is combined into the composite therefore the data that is contained in the subsample is averaged with all other subsamples. The lack of a measure of the sampling error is the cause of the problem confronting the statistician. Multiple samples taken at each location would avoid this problem but costs usually preclude this. A compromise is possible by only analyzing duplicates or triplicates at a percentage of the locations. The exact location is chosen by use of a random number table and should be identified before the study begins. The duplicates should not be made up of a second set of subsamples.

Large cores such as those collect by split spoon can be split lengthwise in half. Each half is thus used as part of two separate composite samples. This avoids the time required to take the second set of cores but provides the duplication necessary for calculating the sampling error.

7.4.2 Compositing with a Mixing Cloth

Soil scientists often use a large plastic or canvas sheet for compositing samples in the field. This method works reasonably well for dry soils but has the potential for cross contamination problems. Organic chemicals can create further problems by reacting with the plastic sheet. Plastic sheeting, however, is inexpensive and can therefore be discarded after each sampling site.

This method is difficult to describe. It can be visualized if the reader will think of this page as a plastic sheet. Powder placed in the center of the sheet can be made to roll over on itself if one corner is carefully pulled up and toward the diagonally opposite corner. This process is done from each corner. The plastic sheet acts the same way on the soil as the paper would on the powder. The soil can be mixed quite well if it is loose. The method does not work on wet or heavy plastic soils. Clods must be broken up before attempting to mix the soil.

After the soil is mixed, it is again spread out on the cloth into a relatively flat pile. The pile is quartered. A small scoop, spoon or spatula is used to collect small samples from each quarter until the desired amount of soil is acquired (this is usually about 250 to 500 grams of soil but can be less if the laboratory desires a smaller sample). This is mixed and placed in the sample container for shipment to the laboratory. The waste material not used in the sample should be disposed of in a safe manner. This is especially important where the presence of highly toxic chemicals is suspected.

7.4.3 Compositing with a Mixing Bowl

An effective field compositing method has been to use large stainless steel mixing bowls. These can be obtained from scientific, restaurant, or hotel supply houses. They can be decontaminated and are able to stand rough handling in the field. Subsamples are placed in the bowls, broken up, then mixed using a large stainless steel scoop. The rounded bottom of the mixing bowl was designed to create a mixing action when the material in it is turned with the scoop. Careful observance of the soil will indicate the completeness of the mixing.]

The soil is spread evenly in the bottom of the bowl after the mixing is complete. The soil is quartered and a small sample taken from each quarter. The subsamples are mixed together to become the sample sent to the laboratory. The excess soil is disposed of as waste.

7.4.4 Laboratory Compositing

Small sets of samples can often be composited better in the laboratory than in the field. A number of the small surface cores discussed in Section 7.1.1 can be placed in the sample bottle for shipment to the laboratory. These can then be placed in a stainless steel laboratory mixer and mixed to the degree needed by the analytical methods. This technique is the only method that may be useful for obtaining composite samples for some types of soils and may be the best method to use if compositing of samples is necessary in a particular situation.

7.5 Replicate Samples

The quality control program will require duplicate or triplicate samples from a percentage of the sampling sites. These may be collected from the composite or they may be comprised of duplicate sets of samples. The latter is the preferred method.

A question often arises about how to handle the analytical data for these multiple results. All analytical results for the field replicates should be reported. Proper statistical designs can use this data to increase the precision of the estimates made.

There is a tendency on the part of many scientists to discard unusual results (outliers) and to average the remainder of the samples. The discussion on soil variability given earlier should point up the problem with this approach. The outliers are probably part of the normal, wide variation seen in soils data. Averaging the numbers in effect throws away data on the sampling error that is needed to determine the reliability of the data collected.

7.6 Miscellaneous Tools

Hand tools such as shovels, trowels, spatulas, scoops and pry bars are helpful for handling a number of the sampling situations. Many of these can be obtained in stainless steel for use in sampling hazardous pollutants. A set of tools should be available for each sampling site where cross contamination is a potential problem. These tools sets can be decontaminated on some type of schedule in order to avoid having to purchase an excessive number of these items.

A hammer, screwdriver and wire brushes are helpful when working the split spoon samplers. the threads on the connectors often get jammed because of soil in them. This soil can be removed with the wire brush. Pipe wrenches are also a necessity as is a pipe vise or a plumbers vise.

7.7 Record Keeping

One of the vital components of the protocol is to adequately define the records required during the study. Good records become extra important if litigation results from the data collected. Every sample will be questioned in an attempt to either discredit or verify the data depending upon the side of the issue the attorney represents. Some of the records are discussed below.

7.7.1 Log Books

The sample teams should maintain an official log book of the investigation. Observations of the field conditions, equipment used, procedures followed and crew members involved are recorded for each day's sampling. These log books should be bound and all data must be recorded in ink (preferably black ink). Each log book should be maintained by the crew leader and signed by him. No erasures are allowed. When mistakes are made the data is lined out with one line only and the corrected data entered above the incorrect entry or on the next line of the log.

7.7.2 Site Description Forms

These serialized forms record the conditions at each site at the time the samples are collected. A sketch map and photographs of the site should be a part of the description. A polaroid-type camera should be used so that the pictures of the sites can be checked before leafing the area of the sample collection. These forms and the back of the photographs should be signed and dated by the crew leader responsible for taking the samples. The KNACK site description form should be used in most cases where the USEPA is involved.

7.7.3 Sample Tags

Tags made up according to the specifications provided by NEIC should be printed for use in the soils study. A tag must be prepared for each sample. All data must be included on the tag at the time the sample is collected. Wet samples should be double bagged with the tag in the outer bag. The person collecting the sample should sign the tag.

7.7.4 Chain-of-Custody Forms

This form is perhaps one of the most important as far as the legality of the samples is concerned. Chain-of-custody traces the possession of the sample from its origin through to data analysis. Most field researchers are not accustomed to observing the care needed to insure the safe custody of their samples. The samples must be in the physical custody of the scientist collecting the sample or else be secured in a facility with controlled, limited access until the samples are signed for and transferred to another responsible party. Samples must not be left unattended in an unlocked vehicle for any reason. There is nothing more disconcerting to technical representatives of the regulatory agencies than to spend hours working with data collected by field teams and then find the data is open to question because the chainof-custody had been violated. Samples are a valuable resource and should be treated accordingly.

7.8 Decontamination

One of the major difficulties with soil sampling arises in the area of cross contamination of samples. The most reliable methods are those that completely isolate one sample from the next. Freshly cleaned or disposable sampling tools, mixing bowls, sample containers etc. are the only way to insure the integrity of the data.

Field decontamination is quite difficult to carry out, but it can be done. Hazardous chemical sampling adds another layer of aggravation to the decontamination procedures. The washing solutions must be collected for disposal at a waste disposal site. The technique outlined below has been used under field conditions.

7.8.1 Laboratory Cleanup of Sample Containers

One of the best containers for soil is in the glass canning jar fitted with Teflon or aluminum foil liners placed between the lid and the top of the jar. These items are cleaned in the laboratory prior to taking them into the field. All containers, liners and small tools should be washed with an appropriate laboratory detergent, rinsed in tap water, rinsed in distilled water and dried in an oven. They are then rinsed in spectrographic grade solvents if the containers are to be used for organic chemical analysis. Those containers used for volatile organics analysis must be baked in a convection oven at 105 °C in order to drive off the rinse solvents.

The Teflon or aluminum foil used for the lid liners is treated in the same fashion as the jars. These lines must not be backed with paper or adhesive.

7.8.2 Field Decontamination

Sample collection tools are cleaned according to the following procedure.

- Washed and scrubbed with tap water using a pressure hose or pressurized stainless steel, fruit tree sprayer.
- Check for adhered organics with a clean laboratory tissue.
- If organics are present, rinse with the waste solvents from below. Discard contaminated solvent by pouring into a waste container for later disposal.
- · Air dry the equipment.
- Double rinse with deionized, distilled water.
- Where organic pollutants are of concern, rinse with spectrographic grade acetone saving the solvent for use in step 3 above.
- Rinse twice in spectrographic grade methylene chloride or hexane, saving the solvent for use in step 3.
- Air dry the equipment.
- Package in plastic bags and/or precleaned aluminum foil.

The distilled water and solvents are flowed over the surfaces of all the tools, bowls etc. The solvent should be collected in some container for disposal. One technique that has proven to be quite effective is to use a large glass or stainless steel funnel as the collector below the tools during flushing. The waste then flows into liter bottles for later disposal (use the empty solvent bottles for this). A mixing bowl can be used as a collection vessel. It is then the last item cleaned in the vessel. It is then the last item cleaned in the sequence of operations.

The solvents used are not readily available. Planning is necessary to insure an adequate supply. The waste rinse solvent can be used to remove organics stuck to the tools. The acetone is used as a drying agent prior to use of the methylene chloride or hexane.

Steam cleaning might prove to be useful in some cases but extreme care must be taken to insure public and worker safety by collecting the wastes. Steam alone will not provide assurance of decontamination The solvents will still have to be used.

7.9 Quality Assurance

Quality assurance in EPA is usually handles by someone other than the sampling team. The field team is responsibility for insuring that the quality assurance program is carried out correctly, however. The team will be required to take duplicate samples at prescribed intervals and will be required to submit field blanks of all materials used. It would be desirable to prepare a bulk soil for use as a field blank for the soil samples. This will have to be handled very carefully because of the difficulty in finding "clean" soil for use as the blank. Distilled water can be used in lieu of a soil blank. Additional samples such as equipment swipes, rinse water and solvents should be taken on a regular basis to verify the quality of the data obtained from the samples. Procedures for handling quality assurance have been outlined in an interim guideline prepared by the EPA Office of Monitoring Systems and Quality Assurance of the Office of Research and Development (OMSQA, 1980).

7.10 Safety

Toxic chemicals create a hazard for the soil sampling team. The team often is operating above plumes containing mixtures of highly toxic chemical. The drillers and excavators are in an especially hazardous position. An industrial safety specialist should be consulted prior to undertaking a study of these highly contaminated areas. Physical examination should be given to the crew on a regular basis unless the sampling team operates only on rare occasions in which case they should have physicals before and after the sampling effort.

Many of the field team members will not want to follow the procedures outlined by the safety office. This should not be tolerated. This problem seems to be especially acute with the drilling crews. Every effort should be made to provide the teams with adequate training on the use of all safety equipment and recovery procedures prior to going into the field.

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