MODULE 8.0: SAMPLING AND MEASUREMENT PRACTICES

Introduction

Welcome to Module 8.0 of the Fuel Cycle Processes Directed Self-Study Course! This is the eighth of nine modules available in this self-study course. The purpose of this module is to provide a basic understanding of the practices of sampling and measurement. This self-study module is designed to assist you in accomplishing the learning objectives listed at the beginning of the module. There are 14 learning objectives in this module. The module has self-check questions to help you assess your understanding of the concepts presented in the module.

Before you Begin

It is recommended that you have access to the following materials:

- Trainee Guide

Complete the following prerequisite(s):

- Module 1.0: Overview of the Nuclear Fuel Cycle

How to Complete this Module

1. Review the learning objectives.
2. Read each section within the module in sequential order.
3. Complete the self-check questions and activities within this module
4. Check off the tracking form as you complete the self-check questions and/or activity within the module.
5. Contact your administrator as prompted for a progress review meeting.
6. Contact your administrator as prompted for any additional materials and/or specific assignments.
7. Complete all assignments related to this module. If no other materials or assignments are given to you by your administrator, you have completed this module.
8. Ensure that you and your administrator have dated and initialed your progress on the tracking form.
9. Go to the Trainee Guide and review the steps for course completion.

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LEARNING OBJECTIVES

8.1 Upon completion of this module, the trainee will be able to describe fundamental concepts of health and safety and abnormal occurrences, and selected engineering controls and preventive measures.

8.1.1 Define the following basic terms associated with sampling and measurement activities:

- Representative
- Analyte
- Population
- Sampling
- Measurement

8.1.2 Identify sampling and measurement issues associated with the following selected components:

- Sample medium
- Sampling and measurement quality criteria
- Quality assurance/quality control
- Standard operating procedures

8.1.3 Identify types of destructive and nondestructive measurements used in fuel cycle facilities.

8.1.4 Identify sources of error in the measurement process.

8.1.5 Identify the following methods of sampling and measurement:

- Grab sample
- Composite sample
- Continuous sample
- Continuous monitoring

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8.1.6 Identify techniques used in sampling liquids.

8.1.7 Identify techniques used in sampling gases.

8.1.8 Identify techniques used in sampling solids.

8.1.9 Identify common sampling and measurement concerns related to sample collection and handling.

8.1.10 Identify measurement program concerns.

8.1.11 Identify sampling and measurement concerns for maintaining nuclear criticality safety.

8.1.12 List applicable regulations and requirements for sampling and measurement of effluents and discharges and within the environs of fuel cycle facilities.

8.1.13 Identify the purpose of the Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM).

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Learning Objective

When you finish this section, you will be able to:

8.1.1 Define the following basic terms associated with sampling and measurement activities:

- Representative
- Analyte
- Population
- Sampling
- Measurement

8.1.2 Identify sampling and measurement issues associated with the following selected components:

- Sample medium
- Sampling and measurement quality criteria
- Quality assurance/quality control
- Standard operating procedures

OVERVIEW OF SAMPLING AND MEASUREMENT

For this course, sampling and measurement are considered for in-process and environmental areas. **In-process** sampling and measurement refer to sampling and measurement of materials that are within the various operations of a nuclear fuel cycle facility. For example, in a conversion plant, uranium oxide concentrate (U_3O_8) is purified to produce uranium hexafluoride (UF_6). The activities that occur during the conversion process are normally referred to as in-process.

The impact that the nuclear fuel cycle has on the environment is another area where sampling and measurement play an important role. **Environmental** sampling and measurement are used to characterize the types and quantities of radiological and nonradiological contaminants contained in effluents and discharges and within the environs of nuclear fuel cycle facilities.

During the previous modules, selected in-process and environmental sampling and measurement activities were noted. This module is not intended to be a repeat of all of the previously mentioned sampling and measurement activities. The intent of this module is to

assist you in defining basic terminology and identifying methods, techniques, problems, and regulations associated with sampling and measurement in fuel cycle facilities.

There are several reasons for in-process and environmental sampling and measurement. Sampling and measurement activities in nuclear fuel cycle facilities are performed to:

- Establish and maintain nuclear material control and accountability on all nuclear materials onsite
- Verify adherence to product specifications
- Verify compliance with relevant federal, state, or local requirements for worker and public safety and health
- Maintain nuclear criticality safety
- Monitor process contaminants

As part of each fuel cycle facility license or certification, a sampling and measurement program should be adequately established and maintained.

Establish and Maintain Nuclear Material Control and Accountability

Facilities develop and implement a nuclear material control and accountability (MC&A) program to ensure that all nuclear materials on inventory have been accurately accounted for, and that diversion of nuclear material has not occurred.

This system of control and accountability provides fuel cycle facilities the means to characterize and quantify all nuclear material contained in-process, storage, and waste streams including both accidental and normal process losses.

Where several processes are occurring within a single facility, sampling and measurement functions are separated into smaller areas known as material balance areas (MBAs). Each MBA samples, measures, and accounts for all nuclear material entering and exiting the MBA.

Note: The NRC has additional courses devoted to material control and accountability. This course was not designed to cover this information even though it is an extremely important part of sampling and measurement in fuel cycle facilities.

Verify Adherence to Product Specifications

Sampling and measurement activities are conducted at each facility to ensure a quality product is delivered to meet specific customer needs. These activities may include receipt or preparation of samples for onsite or offsite analysis. Facility operations personnel may also verify adherence to customer product specifications at any stage in the development of the product. The extent of verification is dependent on facility and customer arrangements.

Verify Compliance With Relevant Federal, State, or Local Requirements

Sampling and measurement are used to determine compliance with applicable standards and permit requirements, assess radiation exposures to members of the public, and assess impact to the environment. Activities include in-process monitoring, routine environmental monitoring, and emergency monitoring procedures.

One aspect of maintaining compliance is related to the protection of personnel, the public, and the environment from unnecessary exposure to facility/site contamination. Operations that are related to this purpose are usually referred to as health physics functions.

Maintain Nuclear Criticality Safety

Sampling and measurement must be conducted to protect against the consequences of an inadvertent nuclear criticality when processing or handling fissionable materials. To maintain a subcritical state, mass and volume, enrichment, geometry or shape, interaction and separation, moderation, reflection, concentration and density, neutron absorbers or poisons, and heterogeneity must be controlled. The combined effects of several of these factors determine whether nuclear material can become critical. Changes made to one of the factors may affect the parameters of the other factors.

Monitor Process Contaminants

Monitoring process contaminants involves characterizing and quantifying foreign or unwanted material. Within a facility, this may be conducted through observation of chemical process variables by means of pressure, temperature, and flow, and may also include the collection and analysis of liquid, gaseous, or solid samples or measurements. In the environs of a facility, this includes the collection and analysis of samples or measurements of liquid, gaseous, or airborne effluents or discharges.

Terminology and Basic Concepts

It is important, at the beginning of this module, to state certain basic concepts and strive to utilize them consistently throughout the course. **Sampling** and **measurement** in the nuclear fuel cycle involves extracting a **representative** portion of an **analyte** of interest from a total **population** and obtaining an indication of its relevant property.

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Terminology definitions:

- **Sampling** is the physical extraction of a prescribed portion of a material for the purposes of measurement.
- **Measurement** is the quantification of the mass, volume, quantity, composition, or other property of a material. It may involve the actual collection of a sample of the material with subsequent analysis or be accomplished in-situ.
- **Representative** means the degree to which sampling and measurement results demonstrate the population characteristic of interest.
- An **analyte** is the chemical for which a sample is analyzed.
- A **population** is the larger aggregate of materials from which a sample is retrieved.
- A **sample** is an extracted portion or subset of a medium (material) of concern.

Applicable Regulations and Requirements

- National Environmental Policy Act (NEPA) P.L. 91-190
- Title 10 CFR Part 20, "Standards for Protection Against Radiation"
- Title 10 CFR Part 40, "Domestic Licensing of Source Material"
- Title 10 CFR Part 50, "Licensing of Production and Utilization Facilities"
- Title 10 CFR Part 70, "Domestic Licensing of Special Nuclear Material"
- Title 10 CFR Part 71, "Packaging and Transportation of Radioactive Materials"
- Title 10 CFR Part 72, "Licensing Requirements for Independent Storage of Spent Nuclear Fuel and High-Level Radioactive Waste"
- Title 10 CFR Part 74, "Material Control and Accounting of Special Nuclear Material"
- Title 10 CFR Part 76, "Certification of Gaseous Diffusion Plants"
- Title 30 CFR Part 828, "Special Permanent Program Performance Standards — In-Situ Processing"
- Title 40 CFR Part 190, "Environmental Standards for Nuclear Power Operations"

ESTABLISHING AND MAINTAINING A SAMPLING AND MEASUREMENT PROGRAM

Acquisition of data sufficient to support sampling and measurement objectives depends largely on project scoping and planning. Through application of a structured development process, project scoping and planning translate sampling and measurement objectives into a **sampling and measurement plan (SMP)** that defines the following activities necessary to acquire data of requisite quality:

- Field
- Laboratory
- Data handling
- Quality assurance

Scope

Scoping and planning efforts are documented in the SMP. The SMP components identified above would constitute the major sections of the plan. The plan should be written to accomplish the following:

- Clearly and unambiguously define the logical basis of the sampling and measurement activities
- Be executable by field and laboratory technicians based on the information contained in the plan
- Allow Nuclear Regulatory Commission (NRC) staff to conduct a meaningful inspection of sampling and measurement activities to determine compliance with license and regulatory requirements

Sample Medium

The sample medium is a substance (e.g., air, water, soil, or other solid material) that serves as the carrier of the analyte(s) of interest. To some extent the sample medium dictates the type of technique used to obtain the sample. Before any samples are collected, it is important to decide what type of sample should be collected. Samples at fuel cycle facilities can be in any of the following forms:

- Liquid
- Gaseous
- Solid

Any liquid with the potential for radioactive contamination should be monitored. The size of the liquid sample needed will be determined by the analytical procedure used and the desired **minimum detectable concentration (MDC)** (the a priori activity level that a specific instrument and technique can be expected to detect 95% of the time) of the radionuclide of concern.

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Evaluating the impact of airborne contaminants necessitates the accurate determination of the concentration of the contaminant present in a unit volume of air. This value is determined either directly from the airstream or following collection of the contaminant from a known volume of air on a suitable medium. The analytical methods for analysis of the material will depend upon the material's physical state (i.e., gas, vapor, or particle), and upon its chemical composition.

The sampling of solids as they occur in nature or as they may be found during some stage of processing is necessary for determination of the following:

- Characterization of isotopic composition and quantity of materials within facility-specific operations
- Process contaminants in streams
- Waste and discharge emissions

Sample media may vary greatly in form. Soil is an example of a solid medium for sampling, as well as the raw materials and product solids at fuel cycle facilities. Sampling may be necessary for many uranium compounds (e.g., UO_2F_2, U_3O_8, UO_2, UO_3,).

The types of containers used and the sampling devices appropriate to each medium are peculiar to the form of the sample.

Sampling and Measurement Quality Criteria

A sampling and measurement program is directed toward providing data of quality and quantity sufficient to support the intended uses. The following quality criteria have been defined for sampling and measurement activities:

- **Accuracy** is a qualitative concept in the statistical treatment of measurement data used to describe the agreement between the central tendency of a set of numbers and their correct value (or the accepted reference value). It is also used to describe the agreement between an individual value and the correct value (or the accepted reference value).
- **Comparability** is a qualitative expression of the confidence with which one data set can be compared with another. Data should be comparable both at a facility and across facilities.

  Comparability is achieved by using standard techniques to collect and analyze samples and report analytical results in appropriate units.

- **Completeness** is defined as the percentage of measurements that is judged to be valid. Completeness goals (i.e., minimum acceptable percentage) should be established for each sampling and measurement effort to ensure that adequate data are collected. Critical samples or measurements should also be identified, and contingency planning be included to ensure their collection. Completeness can be compromised by problems that preclude or compromise the actual field collection, as well as those that produce invalid or unusable analytical results.
- **Precision** is the degree of agreement of repeated measurements of the same property. A precision statement is derived from the estimated standard deviation of a series of measurements and is expressed as a coefficient of variation.
- **Representativeness** expresses the degree to which sampling and measurement results demonstrate the population characteristic of interest. It is a qualitative criterion that is most closely related to the design of the sampling and measurement program. Factors that influence representativeness define the what, when, and where of the sampling and measurement process and include those that influence accuracy and precision. Representativeness can best be ensured by basing the sampling and measurement process on a well-defined conceptualization of the system or entity being characterized.
- **Reproducibility** is the degree of precision of a laboratory in obtaining the same measurement values of an original sample.

Data Management and Record Keeping

A plan for **data management** and all related activities should be developed as part of the SMP. Elements of this plan should include the following, as appropriate:

- Database design
- Data collection forms
- Data entry
- Data encoding
- Data retrieval and manipulation
- Data summarization
- Data distribution (internal and external)
- Computer hardware and software
- Data security

A system for **documents and records management** is needed. This system should document field and laboratory measurement activities, including databases resulting from these efforts, and is essential to establishing the validity of the measurement process.

A records management plan should be provided that addresses the following elements:

- Records control process
- Document archive and index
- Storage, preservation, and safekeeping
- Document and records accessibility
- Final disposition

All quality-related data operations should be properly documented. Such documentation is particularly important to NRC staff charged with inspection of sampling and measurement activities, since many important activities will be conducted in their absence, and the documentation provides the only evidence of these operations. Types of records of sampling and measurement activities include the following:

- Personnel training records
- Field and in-plant logs
- Field quality control records
- Field and in-plant raw data sheets
- Chain-of-custody records
- Laboratory notebooks
- Raw laboratory data (including instrument background and blanks)
- Laboratory quality control records
- Data validation and reduction reports
- Software verification reports
- Audit and surveillance results
- Calibration data

Table 8-1, Types of Site Sampling and Measurement Records, describes possible documentation relating sampling and measurement programs. Records should be maintained and be accessible to NRC staff as defined in the license or certification.

Table 8-1. Types of Site Sampling and Measurement Records

Document Control Identifiers and Headers	Numbered
Sampling and measurement plan	Sample identification documents
Quality assurance plan	Tags
Analytical forms	Chain-of-custody documents
Logbooks	
Field data records and forms	
Shipping forms	
Correspondence	
Photographs, maps, drawings, etc.	
Checkout logs	
Litigation documents	
Reports: monthly, interim, final	

Quality Assurance/Quality Control

NRC defines **quality assurance (QA)** as comprising all those planned and systematic actions necessary to provide adequate confidence in the results of a measurement program. **Quality control (QC)** is recognized as that component of QA implemented to provide a means to control and measure the characteristics of measurement equipment and processes to establish measurement requirements.

Rigorous implementation and documentation of a comprehensive QA program provide a measure of confidence that the sampling and measurement results are valid. Deficiencies identified in the sampling and measurement processes (through auditing and surveillance) are reported to those responsible for these operations so corrective actions can be taken.

An SMP QA program consistent with the American National Standards Institute (ANSI)/American Society for Quality Control (ASQC) Nuclear Quality Assurance (NQA)-1 quality standard should be developed to ensure that the measurement data collected are sufficient to meet their intended uses. The level of QA appropriate to a particular SMP should be consistent with the importance of the activity.

NRC recognizes that errors can be introduced at any stage of the sampling and measurement process, from sampling to reporting of results, and that the QA program should be applied to all these stages.

Standard Operating Procedures

Procedures and instructions for analytical and data operations, including various quality-related activities, must be documented as a set of step-by-step instructions. These **standard operating procedures (SOPs)** are the primary way the quality of each sampling and measurement activity is controlled and data comparability ensured. SOPs are often included as an appendix to the SMP.

Procedures should be developed for the following general categories of activities:

- Sample collection
- Field and laboratory measurements
- Sample labeling
- Chain-of-custody forms
- Records management
- Packaging, shipment, and receipt of samples for on-site and off-site analysis
- Field and laboratory measurement instrument calibration
- Reduction, evaluation, and reporting of data
- Equipment decontamination
- Training programs for measurement and calibration personnel
- Health and safety
- Disposal of samples (if applicable)

The individual(s) responsible for development and/or approval of these procedures should be experienced in procedure development.

Within SOPs those elements that are relevant should be included; for example, a sample collection SOP would include the following:

- Description of containers for sample collection, preservation, transport, and storage
- Preparation of sample containers
- Sample holding times
- Sample numbering (i.e., coding) scheme
- Sample preservation methods

This information is best summarized in tabular and schematic form to provide implementing personnel with useful aids for assembling these elements. QA requires detailed, step-by-step methods and techniques that address these elements and are best documented as SOPs to control measurement system activities and ensure consistency in application within and across programs. In these tabular summaries, these SOPs can be referenced by number.

Example 8-1 illustrates a field data collection form.

Example 8-1. Sample Record Form

Collection date/time_____/_____Log number 87-1403_____
Collected by_____Others present_____
Sample location_____
(verbal description) _____
Survey station: Grid_____Site_____Position_____

Requestor_____
Property owner/address_____
Legal description_____
Authorization to enter property (if different from above), name/address
_____

Purpose of sampling: Work plan number_____or description_____
_____
_____

Sample type: [ ] Soil, rock, sediment (see attached sheet)
[ ] a/ Vegetation Paired soil sample____
[ ] b/ Animal Processing
[ ] c/ Water [ ] as received
[ ] d/ Air [ ] processed
[ ] e/ Other [ ] other
Sample description (e.g., a/ spinach - vegetative tops)
_____

Type of container: Glass w/Teflon liner [ ], Glass w/Teflon septum [ ],
Ziploc bag [ ], Plastic [ ], Other [ ] - if other describe_____
_____ Volume of sample_____

Analyze for: HG [ ] Multielement [ ] Other [ ]
Logged in laboratory [ ] Reviewed by/date _____
Log number 87-1403 Sample analysis_____
Elements: [ ] mercury [ ] uranium [ ] PCB [ ] other
Analyzed by_____on _____
Procedure used_____
Sample results _____
Checked by_____ on_____
Reported to_____ on_____
Log # Laid out Processed Date analyzed ppm
Blank sand_____ _____ ____ QC number _____ ___ ___
Calculations:

Sample custody must be ensured at all times. Assurance of data quality requires that data be traceable from design to final disposition in the project database. In general, a sample or evidence file is under custody if the sample has the following characteristics:

- In possession of the responsible party
- In view of the responsible party after receipt
- Placed in a secure area by the responsible party
- Residing in a designated, secure area

A verifiable chain of custody must be established for the following components of the measurement program:

- Sample custody in the field
- Transfer of custody and shipment
- Laboratory custody procedures
- Sample storage and security
- Sample archiving and disposal
- Data transfer
- Final evidence files (i.e., records control and storage)

A key to maintaining sample custody is proper sample labeling. Example 8-2 illustrates a typical sample label.

Example 8-2. Typical Sample Label

_____

Collector_____ Sample No._____

Place of Collection_____

_____

Date Sampled _____ Time Sampled _____

Field Information_____

Sample Description _____

_____

_____

Example 8-3 illustrates a chain-of-custody form used to record the chain of custody for field samples and laboratory activities.

Example 8-3. Chain-of-Custody Record

SAMPLE NUMBER	SAMPLING LOCATION	DATE	TIME	COLLECTED BY	REMARKS (INCLUDING CONTAINER AND RELATED INFORMATION)

CUSTODY LOG (NAME, DATE, TIME)

(1) FIELD: ACCEPTED BY _____ RELINQUISHED _____

(2) TRANSPORT: ACCEPTED _____ RELINQUISHED _____

(3) LABORATORY: ACCEPTED _____ RELINQUISHED _____

(4) " _____ " _____

(5) " _____ " _____

(6) " _____ " _____

(7) OTHER " _____ " _____

Self-Check Questions 8-1

NSTRUCTIONS: Complete the question. Answers are located in the Trainee Guide.

1. What are the five reasons for performing sampling and measurement activities in nuclear fuel cycle facilities?

Sampling and measurement activities in nuclear fuel cycle facilities are performed to:

Match the following list of terms in column A to the definitions in column B. Answers are located in the Trainee Guide.

Column A **Terms**	**Column B** **Definitions**
A. Analyte	2._____ The degree to which sampling and measurement results demonstrate the population characteristic of interest.
B. Measurement	3._____The larger aggregate of materials from which a sample is retrieved.
C. Population	4._____The chemical for which a sample is analyzed.
D. Representative	5._____An extracted portion or subset of a medium (material) of concern.
E. Sample	6._____The quantification of the mass, volume, quantity, composition, or other property of a material.
F. Sampling	7._____The physical extraction of a prescribed portion of a material for the purpose of measurement.

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Fill in the missing words in each statement. Answers are located in the answer key section of the Trainee Guide. Choose from the following words:

accessible	contaminants	minimum detectable concentration
accuracy	control	NRC
assurance	custody	planning
characterization	documented	procedures
chemical composition	form	representativeness
comparability	labeling	sample medium
		sampling and measurement plan

8. Acquisition of data sufficient to support sampling and measurement objectives depends largely on project scoping and _____.

9. Scoping and planning efforts are documented in a _____.

10. A sampling and measurement plan should be written to allow _____ staff to conduct a meaningful inspection of sampling and measurement activities to determine compliance with license and regulatory requirements.

11. The _____ is a substance that serves as the carrier of the analyte(s) of interest.

12. The size of the liquid sample needed will be determined by the analytical procedure used and the desired _____of the radionuclide of concern.

13. The analytical methods for analysis of the material will depend upon the material's physical state and upon its _____.

14. The sampling of solids as they occur in nature or as they may be found during some stage of processing is necessary for determination of_____of isotopic composition and quantity of materials within facility-specific operations, process _____in streams, and waste and discharge emissions.

15. The types of containers used and the sampling devices appropriate to each medium are peculiar to the _____ of the sample.

16. Quality criteria for sampling and measurement activities include _____ _____, completeness, precision, _____, and reproducibility.

17. All quality-related data operations should be properly _____ .

18. Records should be maintained and be _____ to NRC staff as defined in the license or certification.

19. Quality_____ comprises all those planned and systematic actions necessary to provide adequate confidence in the results of a measurement program.

20. Quality _____ is recognized as that component of quality assurance implemented to provide a means to control and measure the characteristics of measurement equipment and processes to establish measurement requirements.

21. Standard operating _____are the primary way the quality of each sampling and measurement activity is controlled and data comparability ensured.

22. Sample _____ must be ensured at all times.

23. A key to maintaining sample custody is proper sample _____.

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

8.1.3 Identify types of destructive and nondestructive measurements used in fuel cycle facilities.

8.1.4 Identify sources of error in the measurement process.

MEASUREMENTS

Once samples are collected, they must be analyzed by qualified individuals using appropriate equipment and procedures. Samples should be submitted to a qualified laboratory for analysis.

Radiochemical measurement may involve sample preparation followed by analysis. **Radiochemistry** is the area of chemistry that is concerned with the study of radioactive substances. Analysis may be a direct measurement of activity (counting) or concentration and/or purification followed by counting. Analysis may be performed by nondestructive or destructive methods. Nondestructive analysis (NDA) is when the chemical and physical properties of that sample remain essentially unaltered. Destructive analysis is when the original sample identity is destroyed during the process. Sample preparation can vary considerably.

Gamma spectroscopy is the measurement and interpretation of electromagnetic spectra arising from either emission or absorption of radiant energy by various substances. Spectroscopy provides identification of what the substance is, while spectrometry is the quantification of how much radioactivity exists in a substance.

Laboratory Analytical Methods

In fuel cycle facilities, the following types of measurements are performed to conduct isotopic analysis. These types of measurements are destructive analytical techniques.

Mass spectrometry is used to determine the relative weight percent of uranium isotopes contained in a sample. A mass spectrometer is a gas or surface ionization analyzer that is commonly used for quantitative evaluation of isotopic ratios relative to a standard of nuclear materials. Evaluation is made by introducing a sample into an ionizing source, accelerating it by an electrostatic and beam bending field, separating it according to isotopic mass, detection, and recording of ion current.

Two types of mass spectrometers are generally used to determine isotopic concentration rates in the uranium fuel cycle:

- The **double standard interpolative mass spectrometer** uses dual UF_6 gas standards. Unknown samples for analysis are converted to UF_6, two known UF_6 standards that typically bracket the unknown sample are introduced in sequence into the mass spectrometer, and measurements are made. These measurements are a function of the mole ratio of U235 to the total of the other isotopes of uranium. Use of these measurement data, together with the known composition of the standards, permit the determination of the isotopic concentration of the sample. This method requires that all non-UF_6 samples be converted to UF_6 for input into the mass spectrometer. The UF_6 is typically contained in Type 1-S cylinders and introduced into the mass spectrometer as a gas. High-purity UF_6 samples may not require further purification; however, if purification is required, one of two methods may be used: treatment using (1) cobalt trifluoride or (2) elemental fluorine.

  This instrument can be used for the complete range of isotopic compositions used in the fuel cycle, providing suitable UF_6 gas standards are available. The effects of any residual memory can be corrected. This method has an accuracy, relative to the standards, of 0.02% or better.

- The multiple-filament surface-ionization mass spectrometric method is used to determine isotopic concentration of uranium samples containing only a few milligrams of uranium. This method requires that the sample of uranium be dissolved in a solution of 0.2N to 2N nitric acid. The filament is dipped into the solution and evaporated to dryness before it is placed into the multiple filament assembly. The relative intensities of the uranium isotopes are measured by using a semiautomatic scanning system. The intensity ratios are corrected to the standard. This method does not require conversion of the sample to UF6, uses only a few micrograms of uranium for each measurement, and requires only one filament standard for the analysis of the entire loader assembly of samples. The instrument can cover the complete range of isotopic compositions without residual memory effects. This spectrometer has an accuracy, relative to the standard, of 0.01% or better.

Analytical Chemistry Uranium Contact and Impurities

The Davies-Gray method determines the grams of uranium per gram of sample. Samples are reduced to an oxide and titrated using National Institute of Standards and Technology (NIST) standard potassium dichromate solution. The accuracy is about 0.02% with reference to a NIST standard. This very precise method of determining the uranium in a sample of homogeneous material is required to establish inventory values. This is a destructive analytical technique in that the original sample identity is destroyed.

Fluorimetry

Fluorimetry is used to determine uranium quantity in low-grade uranium waste in concentrations of up to 1%. Extensive sample preparation is necessary to remove elements that inhibit the fluorescence of uranium. The result of this method on samples containing up to 0.05% of uranium is about 10% relative.

Spectrographic Analysis

Spectrographic Analysis is used to determine the relative abundance of elemental materials contained in a sample with reference to a known standard. It is used to determine the presence of impurities and/or neutron poisons contained in uranium prior to conversion and/or enrichment.

Note: Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) is also used for impurities.

Nondestructive Assay Methods

In general, NDA is preferred to measure the enrichment and/or the total U235 contained in a heterogeneous matrix in sample containers up to a 55-gallon drum. Since obtaining a representative sample from this type of material is highly unlikely, short of dissolving and homogenizing the entire drum of material, NDA can provide the most accurate measurement of the U235 in the drum. NDA does not destroy sample mass or identity.

Active Neutron Measurements

The **differential die-away system** uses 14-MeV neutrons generated by a neutron generator to interrogate the entire drum of nuclear material and measure the fast fission neutrons from U235 on a time correlation basis. The system is sensitive to 300 mg/U235 contained in a 55-gallon drum. The 14-MeV neutrons are highly penetrative. A measurement of U235 content is obtained. This system can also be used in a passive mode by powering down the generator. Personnel radiation exposure is minimized, since radiation is produced only when the generator is in operation. Operational history has shown the generator to last about five years in full-time use. This system is stationary; materials are moved to the instrument location for measurement. Known standards are developed to ensure precision and accuracy of the instrument. Interferences consist of unknown quantities of moderating or neutron poison materials.

The californium shuffler system uses a large Cf252 source to induce fissions of U235 in the sample. The sensitivity is about the same amount as the 300 mg of U235 contained in a 55-gallon drum. The high-energy neutrons from the Cf source are used to interrogate the drum of nuclear materials and induce fission in the U235 fissile material.

The Cf source is spiraled around the sample very rapidly and returned to the source holder. Delayed fission neutrons are measured by coincidence or time correlation techniques. Personnel safety during operation is provided by maintaining a safety zone around the instrument. A large poly enclosure is used to house and shield the source. Due to its 2.6-year

half-life, the source must be renewed or replaced about every three to five years. This system is stationary; the materials are moved to the instrument for measurement. Passive neutron counting can also be used.

Passive Neutron Measurements

Passive neutron measurements of nuclear materials can be made by counting the F(a,n) neutrons [chemical source in this reaction would be the presence of fluorine compounds in holdup; reacting with or absorbing an alpha particle and producing or subsequently emitting a neutron] produced primarily from U234 or spontaneous fission neutrons from U235. Gross counting is generally used, but time correlation can be used for samples displaying a high count rate. The detectors are generally He^3 tubes imbedded in high-density poly with a cadmium shield to absorb thermal neutrons. The detectors range from two small He^3 tubes to multiple banks of up to three feet in length. Detector and moderator weight varies from about 50 pounds to several hundred pounds. The instruments are portable or movable on carts or lifts and battery-operated and can be moved to the sample location for measurements. Digital readouts range from a simple count-rate meter for gross counting to sophisticated shift registers for time correlation analysis.

Gamma Ray Measurements

Gamma ray measurements made for material control and accountability are generally made with a high-purity germanium (Hp-Ge) detector coupled to an analyzer. To compensate for sample attenuation and container absorption, when determining total U235, a transmission source is typically used. For massive metal or containers of uranium compounds, the 185.72-KeV gamma ray is absorbed in a few millimeters of sample; therefore, total U235 cannot be determined but enrichment can. The advantage of a high-purity germanium detector is a very high resolution, typically 1 KeV for the 186-KeV gamma ray. The disadvantage is that it requires liquid nitrogen cooling while in use.

Sodium iodide (NaI)Tl detectors can be used with the same instrumentation but suffer from relatively poor resolution (generally about 40 KeV for the 186-KeV gamma ray); however, they have very high sensitivities, do not require cooling, and are relatively inexpensive. Five-inch-diameter NaI detectors are ideal for scanning piping and equipment for uranium holdup. In enrichment plants (where holdups consist mainly of fluoride compounds), shielded NaI detectors coupled to digital readouts are used to locate areas of uranium holdup, then passive neutron detectors are used to quantify the U235 contained in these areas. All NDA equipment requires known standards for calibration.

Gamma ray instrumentation varies from simple digital counting meters to sophisticated computer-operated segmented gamma scanners (SGSs), using transmission source correction while rotating and vertically spiraling the container. The detector and source are collimated to view a vertical span of about two inches on each spiral. Containers may vary from a five-inch diameter container to a 55-gallon drum. Total U235 can be determined if there is limited material attenuation. This instrument is typically used for measurement of uranium contained

in low Z matrices, such as uranium-contaminated chemical trap materials, contaminated pump oils, contaminated clothing and shoe scruffs, mop heads, and other waste materials, to an accuracy of about 5% relative uncertainty. The SGS is a stationary instrument; the sample must be moved to the instrument for measurement.

The Hp-Ge and NaI detectors can be used with portable battery-operated multichannel analyzers or laptop computers equipped with multichannel analyzer (MCA) boards to perform accurate field measurements. They are ideal for verification of UF_6 enrichment in steel cylinders. Typical accuracies for low-enriched uranium is about 5% relative uncertainty when ultrasonic cylinder wall thickness corrections are made.

Environmental, Health, and Safety Instrumentation and Materials to Analyze

Table 8-2 contains descriptions of selected environmental, health, and safety instrumentation, a description of the instruments, and identification of materials that they analyze.

Table 8-2. Selected Environmental, Health, and Safety Instrumentation

Counting Instrument	Description	Materials to Analyze
Alpha Hand-Held Monitor	An alpha hand-held monitor is an oblong probe with a thin Mylar window.	It is used to detect alpha contamination deposited on surface areas.
Alpha Spectrometer	Analysis for specific alpha emitters often requires chemical separation and purification, followed by alpha spectroscopy counting. These analyses usually require an internal tracer to determine chemical recovery and give quantitative results.	Alpha spectroscopy can be used to identify the presence of isotopes of uranium, thorium, and other actinides contained in air filter samples.
Beta/Gamma Pancake Detectors	These detectors are useful for scanning equipment, frisking personnel for contaminated clothing, and scanning personnel work areas for uranium contamination. They are coupled to digital readouts.	These detectors are used to measure gross beta/gamma radiation per square centimeter of detector area.
Gas Proportional Counting	The gas proportional counting method is used to screen a variety of matrices for high alpha and beta nuclides in water and air filters. These detectors do not provide specific isotopic data but can differentiate between alpha and beta particles.	This instrument is used to quantify Sr89/90, Ra228, and Tc99 after chemical separation. It is generally used for many alpha and beta emitters.
Gross Alpha/Beta Counters	Gross alpha/beta counters include Geiger-Mueller gas-filled detectors coupled to simple digital readout battery-powered, portable instrumentation. These detectors do not provide pulse height discrimination of detected radiation.	They are used to measure gross alpha and beta radiation or beta radiation by shielding the detector. This detector is not very sensitive to gamma radiation.
Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)	ICP-MS can be used for the multielement analysis of materials in gaseous, liquid, powder, or solid physical form.	This is generally useful for measuring radioisotopes with relatively long half-lives, such as Th^{230} and U^{238}.
Liquid Scintillation Counting (LSC)	A liquid scintillant coupled to a light-sensitive phototube detects light flashes of a specific wavelength. This generates a pulse, which is amplified and counted by digital electronics.	Liquid scintillation counting is typically used to measure low-energy beta emitters, such as tritium, carbon-14, and nickel-63.

Instrument and Equipment Calibration

Instrument and equipment calibration is one of the primary methods of maintaining measurement accuracy and should be identified for each parameter measured in either the field or laboratory. Calibration must be conducted, using SOPs and standard reference materials (for example, U235 sources or special sources for NDA instrumentation) that are traceable to NIST or other certified reference materials. These include both instrument and mass calibration standards.

Procedures for preparing working standards from certified standard solutions should be developed, and the preparation activities should be recorded. For field instruments, calibration procedures are often provided with the instrumentation. For measurements of nuclear materials by NDA methods, soils of enriched uranium or containers of low-enriched U_3O_8 are generally used. Source strength, source-to-detector distance, detector efficiency, source attenuation, and container absorption are all considered in instrument calibration.

For radiological measurements, reference standards are used to determine counting efficiencies for specific radionuclides or as a function of source energy. The counting efficiency value is used to convert a sample counting rate to activity and/or concentration per unit quantity.

In addition to specifying the method of calibration, the frequency should be identified. Calibration activities should be rigorously recorded.

The performance of the instrument should be checked on a regular basis specified in the QA SOPs.

Note: Health physics equipment and instrumentation are generally calibrated and rechecked. Nondestructive analysis measuring equipment is calibrated by the user for performing specific measurements. Known uranium enrichment standards are used to calibrate the instrument prior to measurement activities.

Data Validation

In a sampling and measurement plan, the principal criteria that will be used to validate the integrity of field and laboratory measurements should be described and include the following information:

- Description of the principal criteria that will be used to validate the field and laboratory measurements
- Fraction of data to be validated
- Limits for acceptability of data sets
- Method for qualifying the quality of reported data

- Standards measurement data

The expected data end use is a factor in determining the type of analysis required for a particular sample. The quality of the data reported is indicated by the following:

- Adherence to approved procedures for sampling and analysis
- Use of appropriate and correct calculations
- Maintenance of adequate records
- QC sample results

A QA program provides integrity and validity to the data.

Data should be reviewed for the following:

- Gross inconsistencies (this would include obvious inconsistencies, such as incorrect units, concentration per liter for a soil sample, or results that are orders of magnitude different from the expected results)
- Use of documented analytical procedures
- Maintenance of critical records
- Appropriate and correct mathematical operations (the data are checked for transcription errors to ensure that the final result applies to the sample that was taken; it is important that calculations are not automatically viewed as being correct data)
- Data clearly traceable to the sample
- Final results that are consistent with all sampling information and analytical data (this provides an analytical value with an associated uncertainty)
- A lower limit of detection (LLD) or minimum detectable concentration (MDC)

Data interpretation is the determination of the quality and meaningfulness of the data.

Quality may be affected by the following:

- Procedural difficulties
- Matrix interferences
- Sample/equipment contamination

These are evaluated by audits and QC samples. Interpretation also involves evaluating theoretical assumptions and possible interferences.

MODULE 8.0: SAMPLING AND MEASUREMENT PRACTICES

For nuclear material control and accountability, the laboratory analyst follows strict analytical procedures and includes measurements standards data. The measurement system instruments and mass determinations are calibrated before use to NIST, NBL, or other recognized national standards.

Analytical data may be ambiguous, misleading, or incorrect. To ensure that data are useful, calculations should be reviewed for possible known interferences, and the limits should be delineated.

Note: Interferences are frequently considered to be compounds whose presence obscures the measurement of the analyte of interest by introducing an unrelated analytical signal where the analyte is measured. They can also suppress the signal of interest to a level at which the analyte cannot be accurately measured. Interferences are chemical or physical properties of samples that cause errors in the measurement process and can increase uncertainty and detection limits. Any analysis has potential interferences. These should be specified in the analytical procedures. When interferences occur, they should be noted with the results. Despite interferences, data may still be considered useful, although limited.

Once data are validated, an assessment of their overall quality in light of the quality criteria (precision, accuracy, and completeness) should be accomplished to ensure that the data will support their intended uses. If statistical procedures are used to assess precision and accuracy, they should be fully described. Limits of acceptability of the data sets should be specified.

All methodologies for manipulating data for analysis and interpretation should be independently verified by checking of the algorithm and computation of a fraction of the data. Data input to the computation routine should be independently verified.

Computer programs used to manage or manipulate project data should be verified by independent evaluation of the algorithm and through standard software engineering (input-output) techniques. The extent of software verification required will depend on whether the software is a commercial, off-the-shelf product or has been written specifically for the SMP.

Verification should occur before initial use and immediately after each program modification. Software documentation should include a description of the algorithm and, if possible, a listing of the code.

One of the key considerations in designing a QA/QC program for a measurement process is determining how many and what type of QA/QC samples are required for an adequate assessment of the data quality.

The percentage of total monitoring effort invested in QA/QC samples depends on the following considerations:

- Size of the project (in general, the smaller the size, the greater the percentage of QA/QC samples)
- Available knowledge concerning sampling and analytical procedures
- Relationship of risk to human health and the environment at various pollutant concentrations
- Nearness of action levels to method detection limits
- Natural variability and distribution of analytes

If data validation or data assessment activities identify unacceptable occurrences of quality problems (nonconformances), corrective actions may be required. Typical corrective actions include the following:

- Retraining of sampling technicians in implementation of SOPs
- Changing an SOP to meet unanticipated conditions
- Equipment decontamination
- More frequent preventive maintenance

SOURCES OF ERROR IN THE MEASUREMENT PROCESS

Error in the sampling and measurement process is the difference between an observed or measured value and its true value. It consists of two components: systematic error and random error.

Systematic Error

A **systematic error** is a condition in which there is a consistent deviation of the results of a measurement process from the actual or true values. The cause for the deviation, or bias, may be known or unknown; however, it is considered assignable (i.e., the cause can be reasonably determined).

Bias, which affects the accuracy of the measurement process, can accumulate during the measurement process when the following elements are inappropriate:

- Sampling design
- Sampling procedure
- Analytical procedure
- Interaction with containers
- Standards
- Inaccurate instrument calibration

In addition, when occurring in a systematic manner (to entire batches of samples) sample contamination, deterioration, and losses (leakages) may cause bias.

Bias introduced in the laboratory is often investigated by using the following:

- Spike samples (to determine percent recovery of the analyte from the medium)
- Measurement of reference standards

Blanks are used to detect both field and laboratory contaminants.

Random Error

Random error refers to variations of repeated measurements made within a sample set that are random and individually not predictable. The causes of random error are assumed to be indeterminate or nonassignable. Since random errors are assumed to be normally distributed, they have equal likelihood of giving measurements above or below the true mean; therefore, they do not affect accuracy. Instead, random errors are the source of imprecision in the measurement process.

Random errors may be introduced into the measurement process in the field during the following:

- Sample collection
- Handling
- Transportation
- Preparation of samples for shipment

In the laboratory, random errors can be introduced during the following:

- Subsampling of the field sample
- Preparation of the sample or subsample for analysis
- Analysis
- Data management activities

Total Measurement Error

The **total measurement error** is the sum of the random and systematic errors associated with field and laboratory activities. In designing QA/QC protocols for a measurement process, the greatest emphasis should be applied to sources that contribute the largest amounts of error.

Sensitivity is the minimum amount of a radionuclide or other material of interest, expressed as a ratio (e.g., percent [%]) or concentration (e.g., parts per million [ppm]) that can repeatedly be detected by an instrument, system, or procedure.

Required Sensitivity and Confidence Level

- **Sensitivity** is a function of sample size, activity, and analytical technique. Usually, the larger the number of samples, the greater confidence one has in the estimate of the true average value.
- **Confidence level**, sometimes referred to as confidence interval, is a value interval that has a designated probability of including some defined parameter of the population. This probability is usually expressed as a percentage. For example, a 95% confidence level indicates that, with 95% confidence, the sample value or mean lies within the confidence interval.

Additionally the following terms relate to errors in measurement:

- **Calibration** is the process of determining the numerical relationship between the observed output of a measurement system and the true value of the characteristics being measured, based on reference standards.
- A **standard** is a material having a known property that can be accurately established based on its physical or chemical characteristics.
- **Traceability** refers to a documented chain of comparisons connecting a working standard to a national standard, such as a standard maintained by the NIST.

Self-Check Questions 8-2

INSTRUCTIONS: Complete the following questions. Answers are located in the Trainee Guide.

Complete the following questions. Answers are located in the Trainee Guide.

1. What is radiochemistry?

2. What is the difference between nondestructive analysis and destructive analysis?

3. What is gamma spectroscopy and how does it differ from spectrometry?

4. Why is mass spectrometry used?

5. What is a mass spectrometer?

6. What are two types of mass spectrometers generally used at uranium fuel cycle facilities?

7. Which type of mass spectrometer mentioned in question 6 requires conversion of the sample to UF6?

8. What analytical chemistry method determines the grams of uranium per gram of sample?

9. Why is fluorimetry used for analysis?

10. Why is spectrographic analysis used?

11. What are two types of active neutron measurements and up to what size of sample container can they measure?

12. What is an advantage of using passive neutron measurements?

13. What is one advantage and one disadvantage of gamma ray measurements?

14. Give an example of how a sodium iodide (NaI)TI detector can be used in an enrichment facility.

15. High-purity germanium and sodium iodide detectors are ideal for verification of what?

16. Calibration must be conducted by using SOPs and what else?

17. What is data interpretation?

Match the following counting instruments listed in column A with the materials to analyze in column B.

Column A **Counting Instrument**	**Column B** **Materials to Analyze**
A. Alpha Hand-Held Monitor	18.____Generally useful for measuring radioisotopes with relatively long half-lives, such as Th230 and U238.
B. Alpha Spectrometer	19.____Used to quantify Sr89/90, Ra228, and Tc99, after chemical separation.
C. Beta/Gamma Pancake Detectors	20.____Used to measure low-energy beta emitters, such as tritium, carbon-14, and nickel-63.
D. Gas Proportional Counting	21.____Used to measure gross alpha and beta radiation or beta radiation by shielding the detector.
E. Gross Alpha/Beta Counters	22.____Used to detect alpha contamination deposited on surface areas.
F. Inductively Coupled Plasma-Mass Spectrometry	23.____Used to measure gross beta/gamma radiation per square centimeter of detector area.
G. Liquid Scintillation Counting	24.____Used to identify the presence of isotopes of uranium, thorium, and other actinides contained in air filter samples.

25. To ensure that data are useful, calculations should be reviewed for possible known interferences, and the limits should be delineated. What are problems related to interferences?

26. When should data be verified?

27. If data validation or data assessment activities identify unacceptable occurrences of quality problems, what typical corrective actions may be required?

28. What is an error in the sampling and measurement process?

29. What is a systematic error?

30. What is a random error?

31. When can random error be introduced into the measurement process in the field?

32. What is total measurement error?

33. What is sensitivity as it relates to measurement?

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

8.1.5 Identify the following methods of sampling and measurement:

- Grab sample
- Composite sample
- Continuous sample
- Continuous monitoring

METHODS OF SAMPLING AND MEASUREMENT

A sample is an extracted portion or subset of a medium (material) of concern. Basically the following methods can be applied when obtaining a sample:

A **grab sample** involves the collection of an instantaneous sample and is appropriate when peak concentrations are sought or when concentrations are relatively constant. A grab sample is representative of a specific location at a given point in time. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease. Sometimes a grab sample will also be referred to as a random sample.

Composite sampling is a number of random or grab samples that are combined into a single sample prior to analysis for the contaminants of concern. To ensure that a composite sample is representative, the individual samples should be proportional to the volume of media represented by each grab sample.

Continuous sampling includes both uninterrupted sampling and repetitive, sequential collection of small samples obtained automatically at intervals short enough to yield a representative sample for the entire sampling period. Continuous samples are removed from pipelines, filling lines, or transfer lines by passage through an area (split or side stream) or medium and are then withdrawn into a sample container of appropriate size for analysis.

Continuous monitoring is the **real-time** measurement of liquid, gaseous, and/or airborne effluents and contaminants, using in-situ measurement systems. Detectors for real-time analysis are direct reading instruments that have been developed to sample and analyze simultaneously.

In-line refers to a system in which a detector or other measuring device is placed in a process of waste stream for purposes of performing measurements.

Finally an, **environmental medium** is a discrete portion of the total environment, biotic or abiotic, that may be sampled or measured directly.

TECHNIQUES

Materials for sampling and measurement may be in liquid, gaseous, or solid form. The types of techniques appropriate for use are particular to the form of the material being sampled. In the next three sections, you will be provided an overview of selected sampling methods for obtaining representative samples from the relevant medium of concern.

Samples generally have to be prepared for testing either individually or as a composite sample. Compositing is used for several purposes, perhaps the most relevant of which is collecting a sample representative of the medium of concern over a specified time period, such as continuous sampling of an effluent stream or continuous high-velocity sampling of ambient air. The methodology for composite sampling must accommodate the variability in the quantity and quality of the medium being sampled. Mixing of the target medium prior to sampling or subsampling is often required to realize a representative sample or subsample for analysis.

Self-Check Questions 8-3

INSTRUCTIONS: Match the terms in column A with their description in column B. Answers are located in the Trainee Guide.

Column A Terms	Column B Description
A. Grab sample	1._____A discrete portion of the total environment, biotic or abiotic, that may be sampled or measured directly.
B. Composite sampling	2._____A system in which a detector or other measuring device is placed in a process of waste stream for purposes of performing measurements.
C. Continuous sampling	3._____The real-time measurement of liquid, gaseous, and/or airborne effluents and contaminants using in-situ measurement systems.
D. Continuous monitoring	4._____Includes both uninterrupted sampling and repetitive, sequential collection of small samples obtained automatically at intervals short enough to yield a representative sample for the entire sampling period.
E. In-line system	5._____A number of random or grab samples that are combined into a single sample prior to analysis for the contaminants of concern.
F. Environmental medium	6._____Involves the collection of an instantaneous sample and is appropriate when peak concentrations are sought or when concentrations are relatively constant.

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

8.1.6 Identify techniques used in sampling liquids.

LIQUIDS

Sampling and measurement of liquids includes:

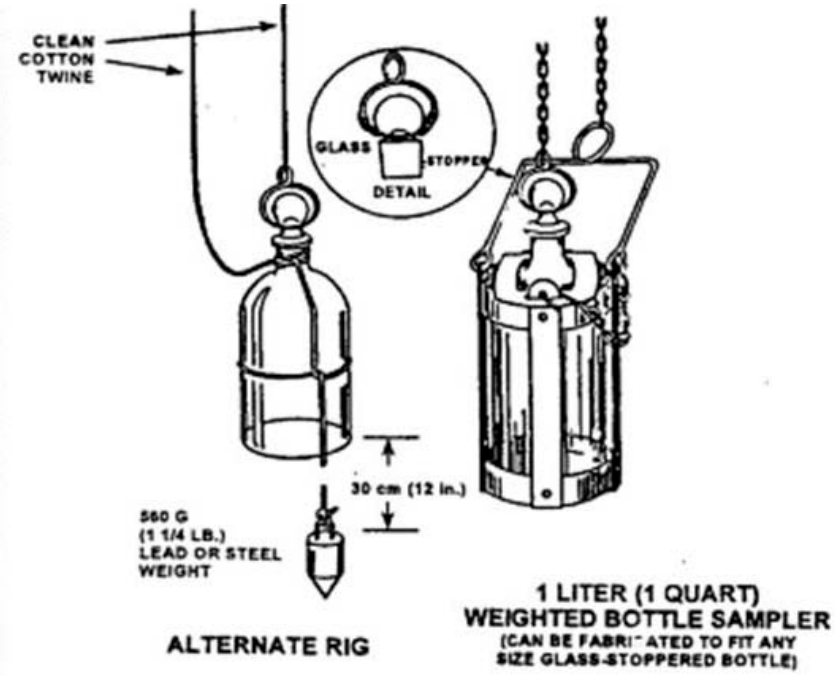
- Sampling liquids in-process
- Liquid effluent/discharges
- Environmental water sampling

SAMPLING LIQUIDS IN-PROCESS

Liquids in Tanks

Bottle sampling is suitable for extracting a grab sample from storage tanks, tank trucks, or tank cars. The bottle can be suspended by twine and weighted with a lead or steel weight. It will generally have a glass stopper, which can be pulled out by another length of twine. For better stability in use and for protection of the bottle against breakage, a weighted carrier suspended by a light chain may be used. See Figure 8-1, Assembly for Bottle Sampling.

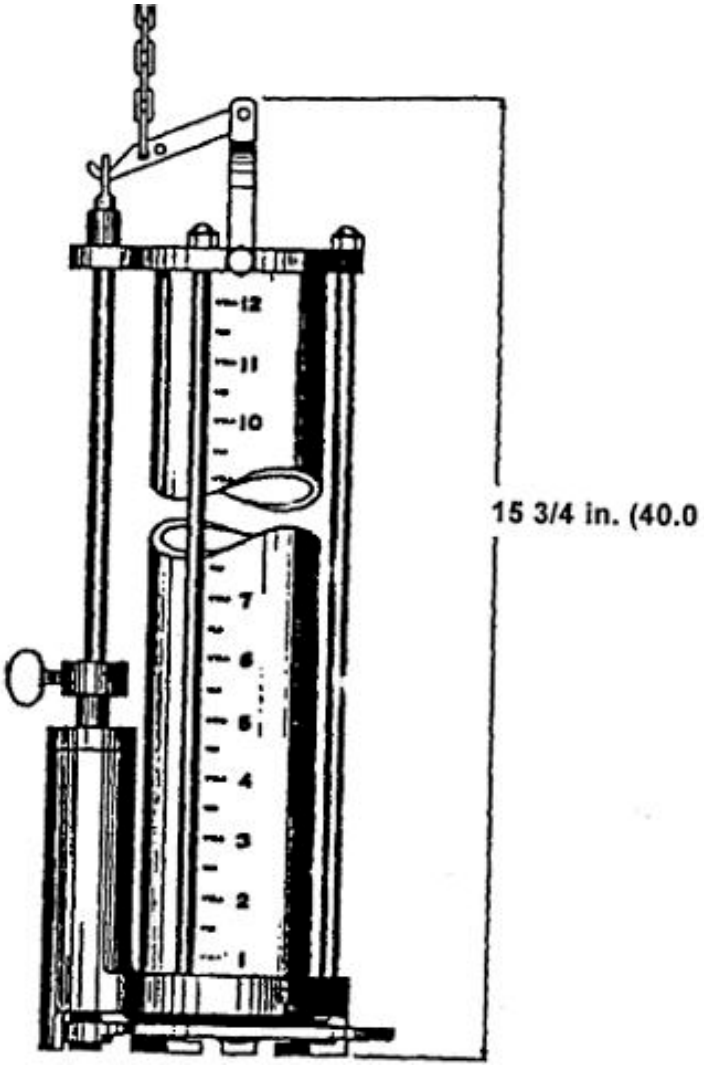
Figure 8-1. Assembly for Bottle Sampling



The sample is withdrawn by lowering the bottle to the desired point, pulling the stopper, and permitting the bottle to fill completely before raising. Separate samples may be obtained at any desired level (i.e., top, six inches below the surface, or upper, middle, and lower samples at respective one-third heights in the tank).

A **thief sampler** may be used to obtain samples from the bottom of tanks. Bottom samples are usually needed to characterize sludges or other material that has settled out. A thief sampler opens when a projecting stem on the valve rod hits the bottom, which opens the container. The sample enters at the bottom, and air is released at the top. The valves close automatically as the thief is withdrawn. A core thief can be lowered to the bottom with valves open to allow flushing of the interior. The valves shut as the thief hits the bottom of the tank. See Figure 8-2, Core Thief, Tap Type.

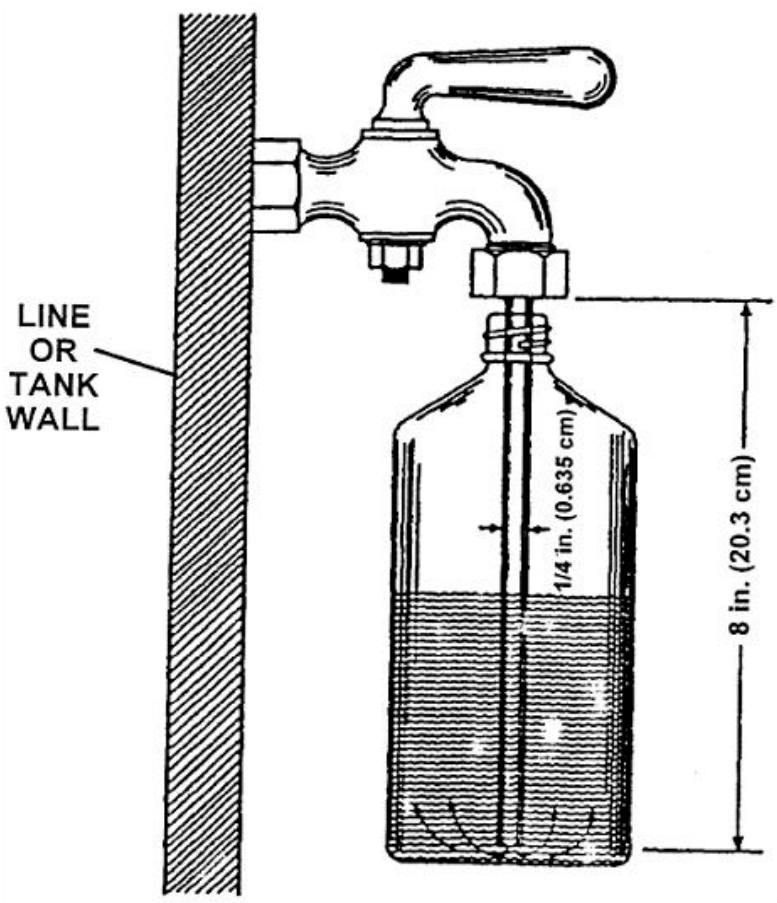
Figure 8-2. Core Thief, Tap Type



Liquids in Tanks Equipped with Taps

Tanks equipped for tap sampling generally have taps placed at several heights (i.e., taps may be located in each third of the tank height) with a 1/4-inch pipe extending inside the tank shell. An open bottle or cup may be used to receive the sample. Separate samples may be drawn from each tap, and a composite sample may be obtained by filling it one-third from each of the taps. See Figure 8-3, Assembly for Tap Sampling.

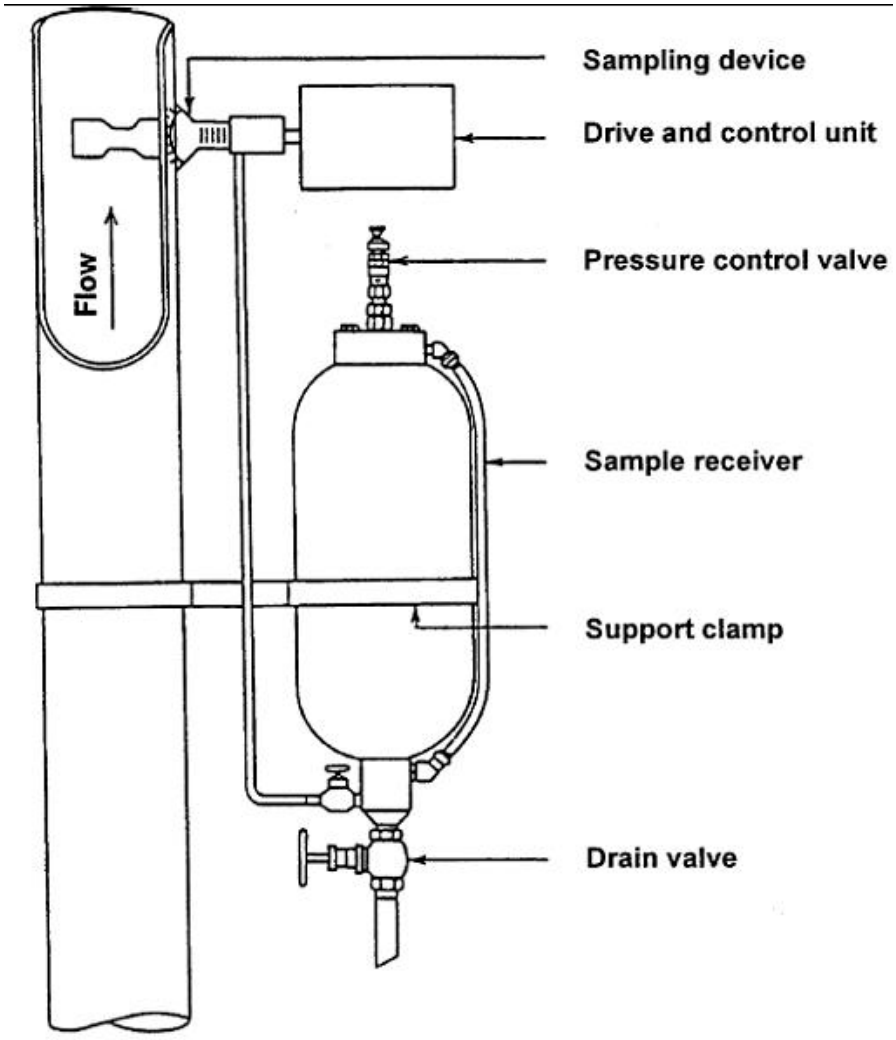
Figure 8-3. Assembly for Tap Sampling



Liquids in Pipelines, Filling Lines, or Transfer Lines

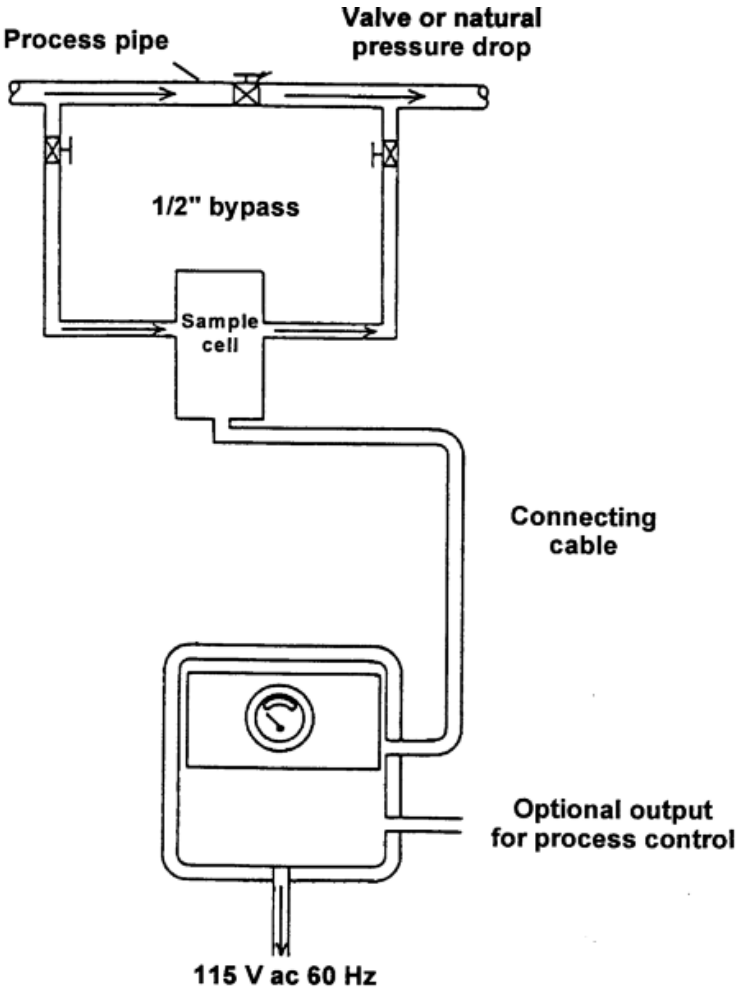
Samples are removed from pipelines, filling lines, or transfer lines by passage through an area (split stream or side stream) or medium and withdrawn into a sample container of appropriate size for analysis. See Figure 8-4, Typical Automatic Sampling Apparatus.

Figure 8-4. Typical Automatic Sampling Apparatus



Sampling for on-stream analysis and control of process liquid or gas mixtures may be accomplished by using a sample cell and instrument installed in a bypass of the main process pipe. See Figure 8-5, Diagram for Continuous Sampling for On-Stream Analysis.

Figure 8-5. Diagram for Continuous Sampling for On-Stream Analysis



Detectors for real-time analysis (direct reading instruments) have been developed to sample and analyze at the same time and may be used during continuous sampling for instant measurements.

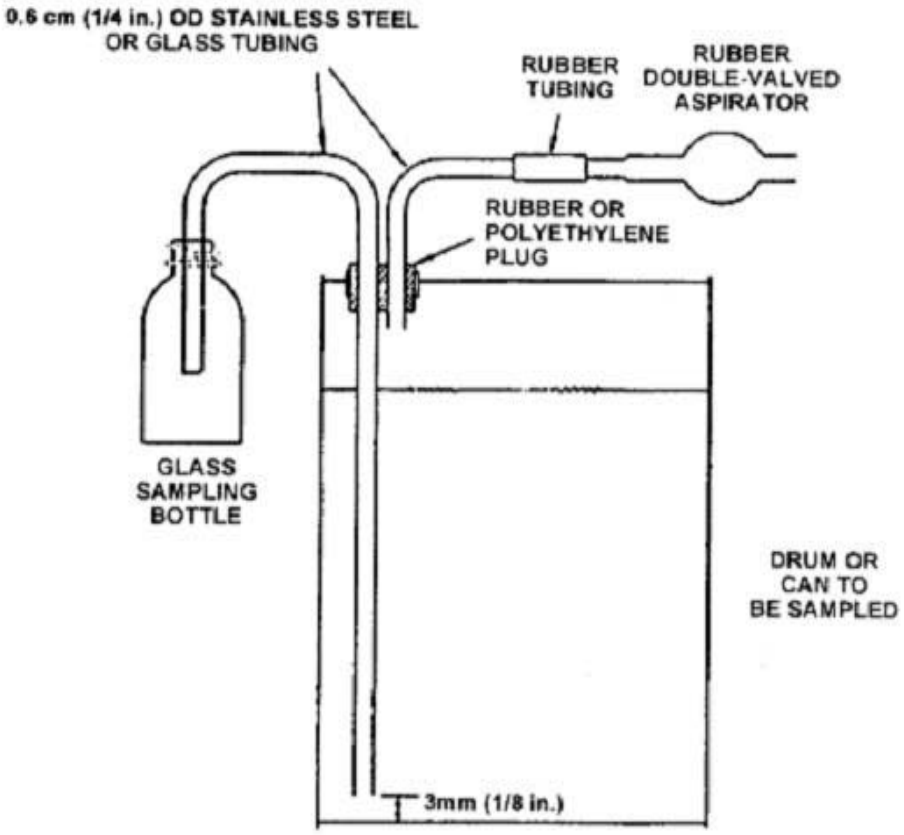
For example, drip sampling uranium solutions in process tanks is a method used to sample solutions of low uranium concentration during transfer to or from process tanks. A special tap is installed in process lines so that a continuous drip sample can be taken while process solutions are being transferred.

Liquids in Drums, Cans, or Bottles

A **dipper** may be used to remove a sample from an open vessel. The dipper may be attached to a stick for the purpose of extending the reach for withdrawing the material being sampled.

Tube sampling can be accomplished by using a pipet for the purpose of extracting a quantity of the material of interest. A sample can be pumped into a container by using a double-valved aspirator bulb and glass or metal tubing, which extends to the desired level in the drum or can. See Figure 8-6, Tube Sampling Assembly.

Figure 8-6. Tube Sampling Assembly



LIQUID EFFLUENT/DISCHARGES

Liquid waste streams from fuel cycle facilities have effluent-stream chemical and biological characteristics that include radioactive, nonradioactive, and sanitary waste. Each is collected, characterized and measured, and treated as needed before release. It is important that the waste streams be characterized for the processes being utilized at the specific site/facility. Waste streams contain both organic and inorganic material, which may be suspended or dissolved in the liquid. Sampling and measurement may be aimed at one or both of these, depending on the process.

Representative samples should be collected at each liquid release point for determination of the quantities and average concentrations of radionuclides discharged in liquid effluents that could reach unrestricted areas, including discharges to a sanitary sewage system. For continuous releases, representative samples should be continually collected at each release point. For batch releases, a representative sample of each batch should be collected.
For some liquid effluents, the licensee may establish, by periodic sampling or other means, that radioactivity in the effluent is insignificant. In such cases, the effluents should be sampled at least quarterly. The licensee should provide information that the samples are representative of actual releases.

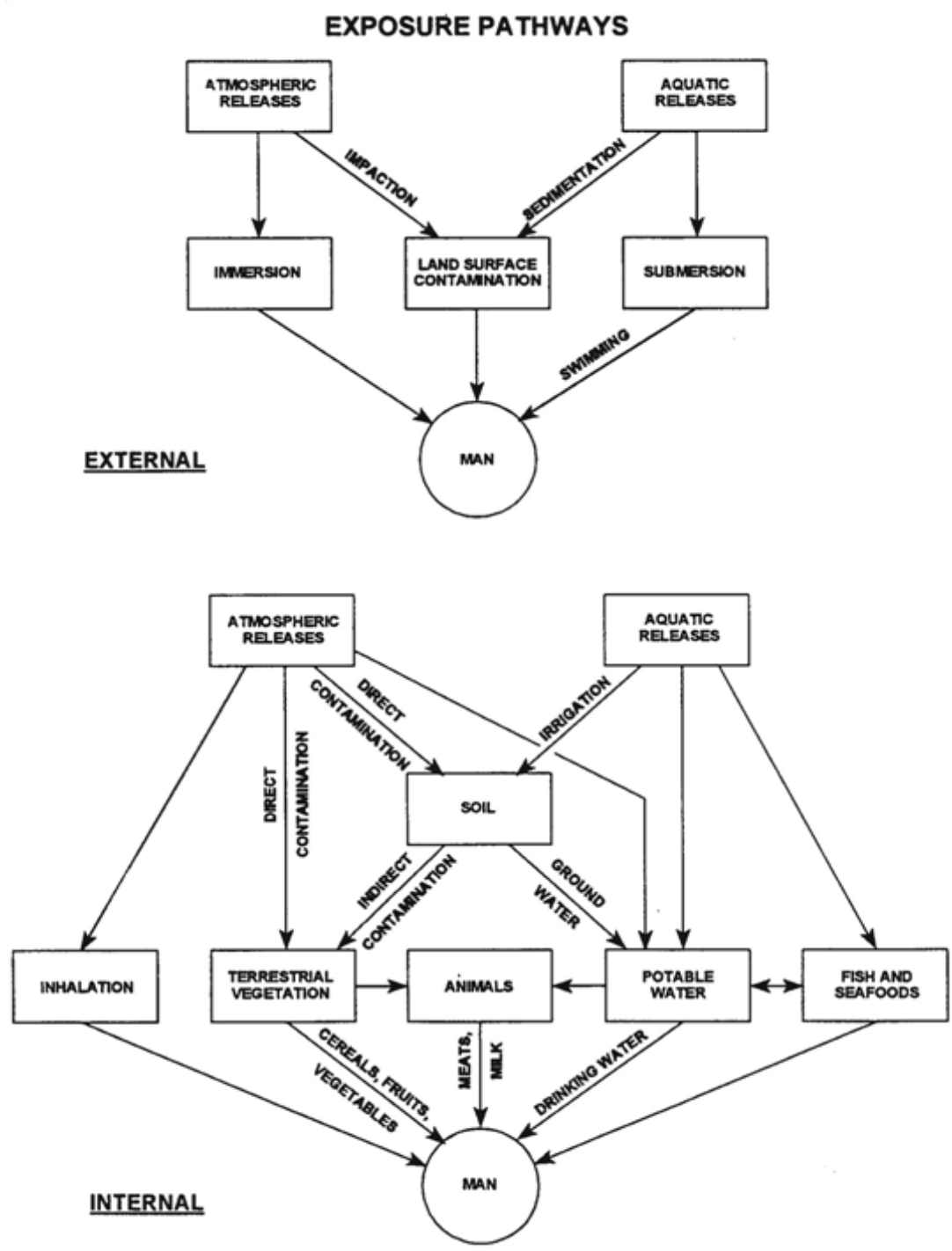
Sampling Systems

A system for sampling and measurement of effluents and discharges from fuel cycle facilities is part of the process developed and engineered to maintain compatibility with the chemical and biological nature of the liquid effluent. Systems are designed to accomplish sampling and measurement accurately and safely and to achieve compliance with applicable standards and regulations. The methods and techniques described previously may be applied during sampling and measurement of effluents/discharges.

Exposure Pathway Analysis

In consideration of sampling design, the significance of the **exposure pathway** should be determined to assist in the proper analysis of effluents/contaminants discharged from the site/facility. The significance is derived by determining the hazard to the environment and the population caused by released materials. See Figure 8-7, Pathways for External and Internal Exposure of Man.

Figure 8-7. Pathways for External and Internal Exposure of Man



The **exposure pathway** is the pathway between the source of released material and intake by individuals. Critical pathways of exposure for population groups living within the vicinity of fuel cycle facilities include the following:

- Airborne effluents
- Contaminated soils, vegetation, and sediments
- Discharged or spilled liquids

The need for surveillance should be evaluated by **exposure pathway analysis** for effluents and contaminants. This analysis should be documented in the site's monitoring plan.

Exposure pathway analysis is impacted by the following:

- Geography - The description of land, sea, air, and the distribution of plant and animal life, including people and their industries
- Demography - The statistical study of human population with regard to density and capacity for expansion or decline

Types of Radiation

In liquid effluent streams, direct measurement is possible only with gamma-emitters or by making gross beta-gamma measurements. In-situ alpha measurement is not feasible. It is important to recognize that the detector chosen should be capable of measuring with the required sensitivity.

High Background

Direct or indirect measurement in areas with high ambient radiation levels (high background) requires shielding or off-line analysis. Off-line monitoring should be used when high radiation dose rates are expected.

Sampling Duration

Liquid effluents in fuel cycle facilities can originate from a fluctuating source or a batch (as opposed to a continuous stream). If the radioactive materials being released are batch materials, it is important that the samples be representative. Prior to release, samples should be withdrawn and analyzed to determine releasability.

Alarms

Alarms should be established to signal the need for corrective actions whenever necessary to prevent public or environmental exposures from exceeding the limits.

Liquid Effluent Monitoring Program

All potentially contaminated liquid effluents generated in the site/facility (except mining and milling) are to be collected in a waste tank(s) prior to release to the environment. A liquid effluent system and flow path should have been developed by the site/facility. All potentially contaminated liquid effluents are batch-released from storage tanks after the tank contents have been sampled and analyzed (prior to release from the sewage treatment system). This provides assurance that concentrations at the point of release are below the NRC 10 CFR Part 20 Appendix B concentration limits and that the water quality meets the state requirements and those of the Environmental Protection Agency (EPA) water quality standards and criteria.

Water from building roof drains, yard drains, and storm water runoff should flow to a holdup basin, where samples are taken following precipitation and analyzed to ensure that any low-level contaminants are below the regulatory limits. These liquids are continuously discharged from a location where the combined liquid flow is continuously sampled for the composite monthly environmental monitoring analyses.

All contaminated liquids should be within established limits prior to release to the sewage treatment system for the site/facility and following treatment and release to unrestricted areas. Additionally, it is recommended that sewage sludge be sampled for possible radionuclide accumulation.

The sampling program should be sufficient to permit determination of the quantities and average concentrations of radionuclides being discharged from the site/facility. The sampling rate at each release point should be sufficient to collect a representative sample of the effluent. The volume of the liquid effluents should also be reported so NRC staff can calculate the quantity of radionuclides discharged.

Liquids in Free or Open-Discharge Streams

A glass or plastic jar with a screw cap may be used to obtain a sample in a free-flowing stream. Portions can be taken at intervals to obtain composite samples representative of the effluent stream.

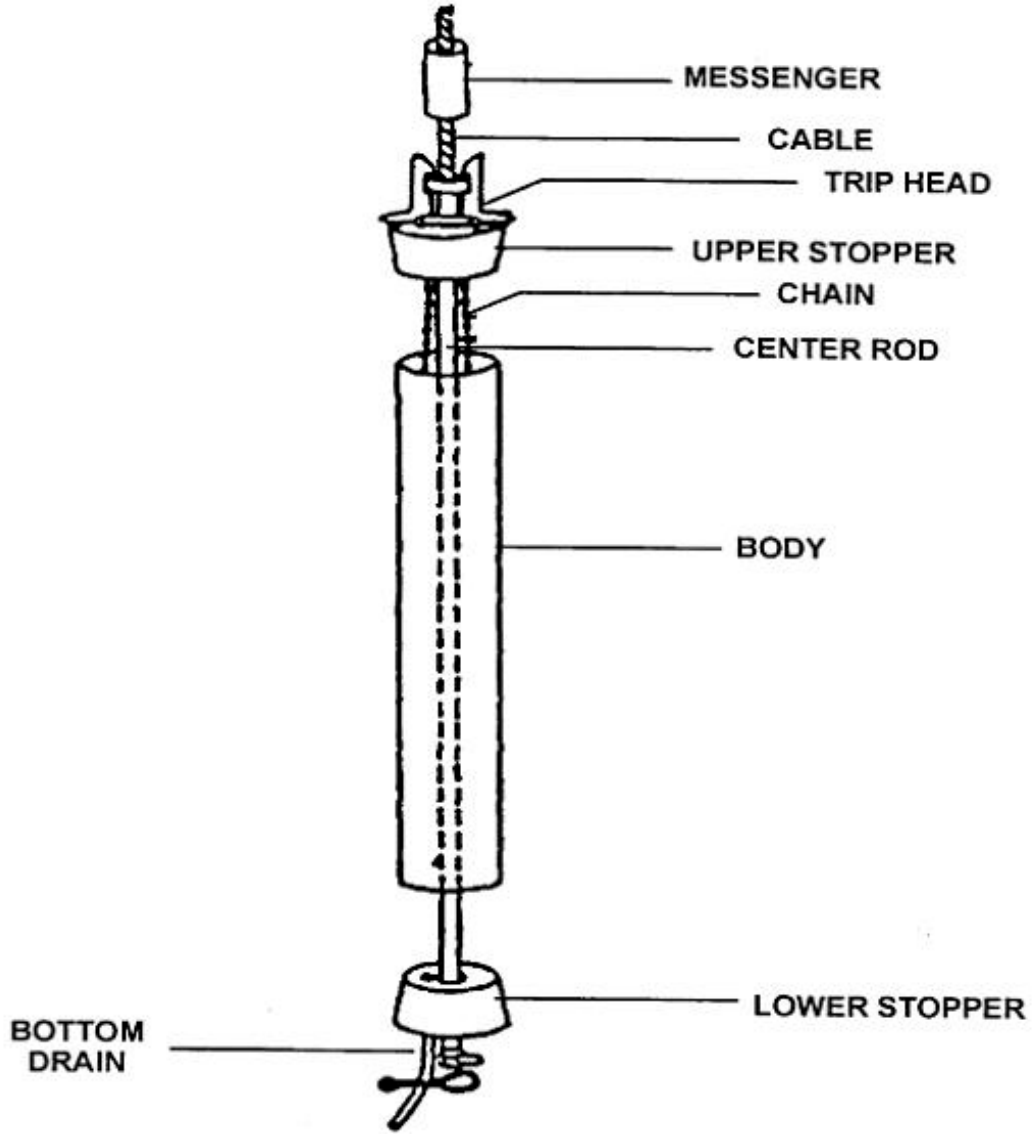
Specialized sampling devices, such as peristaltic tubing pumps, may be used for sampling wastewater in discharge and sewerage systems. There are also devices that collect fixed-size or flow proportional samples on a timed basis and composite them in a collection bottle.

Liquids in Open Bodies

Collecting discrete samples at various depths in shallow water can be accomplished by using a Niskin or Kemmerer sampler. Kemmerer samplers are frequently used to obtain liquid samples from a specific depth. The supporting cable is marked to identify the depth at which the sample will be taken. As it is lowered, the liquid passes freely through the body of the sampler. At the designated depth, a weighted messenger is dropped down the cable. When it reaches

the sampler, it plugs the top and trips a mechanism that closes a plug/stopper at the bottom. Thus, the sampler is sealed and the closed sample tube withdrawn from the body of liquid. To empty the device, the bottom plug is held closed and the upper stopper opened. The sample can then be poured out of the top, capturing a volume of liquid from the appropriate depth. See Figure 8-8, Kemmerer Sampler.

Figure 8-8. Kemmerer Sampler



Special Problems of Sampling Liquids; Suspensions and Hazardous Materials

Suspensions and hazardous materials present special problems in sampling. When sampling suspended solids, the equipment and procedures are much the same as for liquids. It is important to sample the upper and middle portions of a tank separately from the bottom sample (foots). An appropriately proportional composite sample of the upper, middle, and foots sections gives a final sample representative of the whole contents of the tank.

Sampling suspensions often require complete mixing before sampling as well as prior to subsampling to obtain a representative sample. When sampling suspensions, it is often important to know whether contaminants are in the dissolved or particulate (i.e., suspended) state. Therefore, after collection and prior to analysis, such samples are often filtered through inert media (i.e., glass fiber filters of 0.45-micron pore size) and the "dissolved" and particulate fractions analyzed separately.

Multiphase liquids (liquids that may occur in either a liquid or gaseous form, depending on chemical considerations) may also require mixing prior to sampling.

Samples of liquefied gases are usually obtained from containers with fitted sampling ports. Extreme caution must be used when obtaining these samples. They are usually collected in a bomb-type container of appropriate size and construction. Feed-line piping and sampling vessels must comply with operating pressure standards.

Many liquids give off vapors that are toxic or flammable, so adequate ventilation and care against ignition are necessary. Eye protection is recommended during sample handling of corrosive liquids. Room for expansion should be considered when filling sample containers.

Slurry Transfer Holdup

Slurry transfer is the movement of a liquid in which solid or semisolid materials are suspended. An example is the transfer of slurries from precipitation operations to separating or drying operations. Gels or sols containing uranium also may be transferred as slurries.

Slurries are transferred from one process vessel to another by methods that are essentially the same as those used to transfer liquids. However, holdup problems are more complex for slurry transfer because of a tendency of the suspended solids to settle out of the carrier liquid. Although different materials exhibit different settling characteristics, a critical velocity exists below which particles begin to settle out. Such settling is most likely to occur at shutdown or when flow rates are reduced because of abnormal operations. In such situations, pumps and valves may act as sites in which solids can accumulate or be trapped.
Cavities and recesses in pumps used to transfer slurries or suspended solids can collect significant quantities of solid material that are difficult to flush out. Transferring material by jets or gas lifts may minimize this difficulty.

When screw conveyors are used to transfer moist pastes, a coarse intermediate cleanout may be necessary for operational reasons, i.e., to prevent subsequent plugging of the process line. Additionally, a more complete cleanout may be needed at the end of each run. Because frequent cleanouts are necessary for operational reasons alone, a paste transfer method necessitating less interruption is desirable.

Visual access should be provided to all surfaces or spaces where material is likely to accumulate; alternatively, clearance should be provided to permit external use of nondestructive assay instruments or Internal probes to detect the presence of special nuclear material or to identify the location of residual material not visually accessible.

For further information regarding design features and characteristics acceptable to NRC staff for minimizing residual holdup of special nuclear material in process equipment see Regulatory Guide 5.25, Design Considerations for Minimizing Residual Holdup of Special Nuclear Material in Equipment for Wet Process Operations.

Special Considerations

Special considerations for sampling liquids:

- Effluent lines are frequently buried in soil; this could create problems with accessibility.
- The sampling and measurement system should be designed to be compatible with the chemical and biological nature of the liquid effluent.
- Fluctuation in flow rate is common.
- Effluent velocity and corrosion can significantly affect in-line sampling and measurement.
- The environment surrounding the sampling systems and effluent lines must be considered for potential contamination.
- Components of the sampling system should be accessible for maintenance.
- When a **24-hour sample** is indicated, care should be taken to ensure the purpose, type of sample, and sampling technique used. This type of sampling may be used to characterize discharges emitted from a site/facility.
- Twenty-four-hour sample analysis may be composited or analyzed separately.

ENVIRONMENTAL WATER SAMPLING

The most important aspect of an environmental water monitoring program is the detection of contamination. Water sampling and measurement are also used to accomplish the following:

- Characterize the water quality
- Demonstrate compliance with appropriate regulations and requirements

- Demonstrate that environmental contaminants are behaving as predicted by mathematical pathway analyses conducted by fuel cycle facilities
- Estimate exposures to the public
- Perform a long-term trend analysis (for projects requiring long- term historical information)
- Determine if effluents or contaminants are migrating offsite or out of a burial trench/pit

Water Sources Sampled

In the environs of fuel cycle facilities, specific effluent sampling is conducted for the following water sources:

- Groundwater
- Drinking water
- Wastes and discharges
- Surface water and runoff

When environmental water samples are taken, consideration must be given to site-specific groundwater and surface water flows. Surface water possesses an air/water interface; groundwater does not. Nevertheless, groundwater forms a continuum with surface water. The locations of sampling sites are often dictated by the objectives of the sampling program.

Groundwater

Groundwater sampling is conducted to provide information about the condition of subsurface water resources in the zone of saturation. Groundwater can potentially be affected by waste stream discharges from fuel cycle operations. Samples are usually obtained from wells through the use of a variety of pumping and filtering devices. Since groundwater is sampled through pumps placed in wells, care and planning should be taken in developing the best locations for the wells and pumps.

Drinking Water

Drinking water may be supplied from surface water or groundwater sources. Sampling requirements are derived for both sources. Drinking water supplied from a source that receives effluents or contamination from fuel cycle sites/facilities should be sampled on a continuous basis at the point of intake and/or the tap for all public supplies that could be affected by discharges.

Surface water drinking sources should be sampled from treated water at the point of maximum concentration of contamination or release and from untreated water.

Groundwater drinking sources should also be sampled. Characteristics of acceptable sampling locations are within close proximity to the site/facility and down gradient from the nearest surface discharge point (creek, pond, lake, or stream).

Wastes and Discharges

Waste management procedures for liquid wastes result in periodic discharges. Ideally a continuous proportional sampling device would be used; however, if there isn't a direct population exposure pathway, this may not be done. A system should have been developed by the site/facility that accomplishes the following:

- Samples and measures for the presence of wastes and discharges in water sources
- Establishment of locations for sampling based on diffusion and transport studies
- Identification of multiple sampling points, established both close to the discharge outfall location and far enough away from the site/facility so little or no influence on the sample content is anticipated

Surface Water

There are three types of surface water:

- Rivers and streams (constantly moving waters)
- Lakes and ponds (that are not constantly moving)
- Storm runoff (can become part of either of the above)

Rivers and Streams

The best locations to sample in rivers and streams are downstream of turbulence (i.e., downstream of falls, white water, eddy currents, etc.), where contaminants are more likely to be well mixed. Background samples are typically taken upstream of the facility discharge.

Collecting representative samples in, or near, estuarine waters can be extremely difficult because of the differences in temperature and density between freshwater and saltwater. This situation is further complicated by variations due to tidal action. It becomes necessary to increase the number and frequency of sample locations and to relate sampling times to tidal conditions.

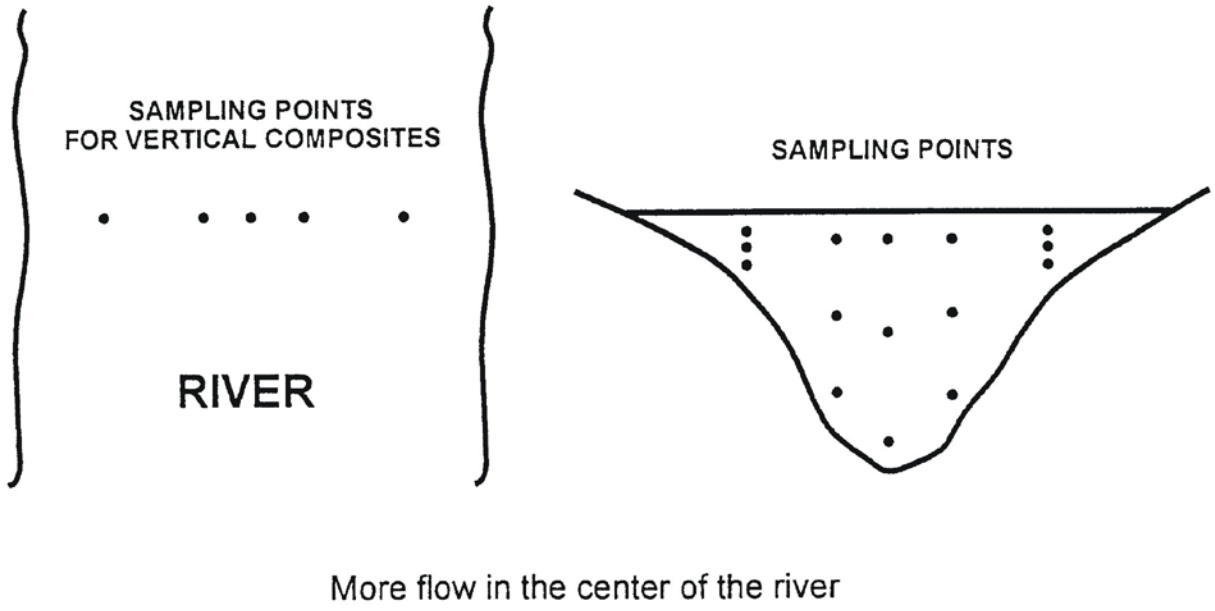
In rivers and streams, mixing is promoted, as streams frequently change direction or meander and exhibit fluctuations in width and depth. Sampling should not be performed immediately downstream of the confluence with tributaries or point sources, such as outfalls of industrial and municipal effluents.

Tests with fluorescent dyes can be performed to identify three to five suitable sampling points. It is often recommended that vertical composites be collected for a sample at a given position along a stream or river. It is more important that sampling points reflect the river's volumetric

flow. Since flow is greater toward the center of a river, the sampling points might be located more toward the center of the river. See Figure 8-9, Water Sampling Points.

If the purpose is to sample sites used as recreational areas or for public water supply intakes, then the sampling sites will be the point of use. A good location for water sampling is often a good location for collecting sediment and fish and making measurements (e.g., a recreational area (park) downstream of the facility). Sampling upstream and downstream for comparison is also appropriate.

Figure 8-9. Water Sampling Points



Lakes and Ponds

Lakes and ponds experience less mixing and have a greater tendency to stratify than streams and rivers. As a result, a larger number of samples will be required. It may be advantageous to establish preferred sampling locations for ponds or lakes. The sampling and measurement of water in lakes and ponds around fuel cycle facilities can provide data that assists in determining the presence and quantity of any effluents or contaminants released from processes into the environment.

Storm Runoff

Storm runoff is important near waste and discharge sites. Runoff, caused by heavy rain, may not follow the normal pathway determined through the pathway analysis that was used in developing the site/facility water sampling plan. Therefore, it is important that the potential for overflow be addressed and monitored to ascertain whether a significant change in the pathway has been created.
For example, contamination from building exhaust and ventilation can build up and contaminate the roof. Runoff from these areas can result in contamination.

ENVIRONMENTAL WATER SAMPLING DEVICES

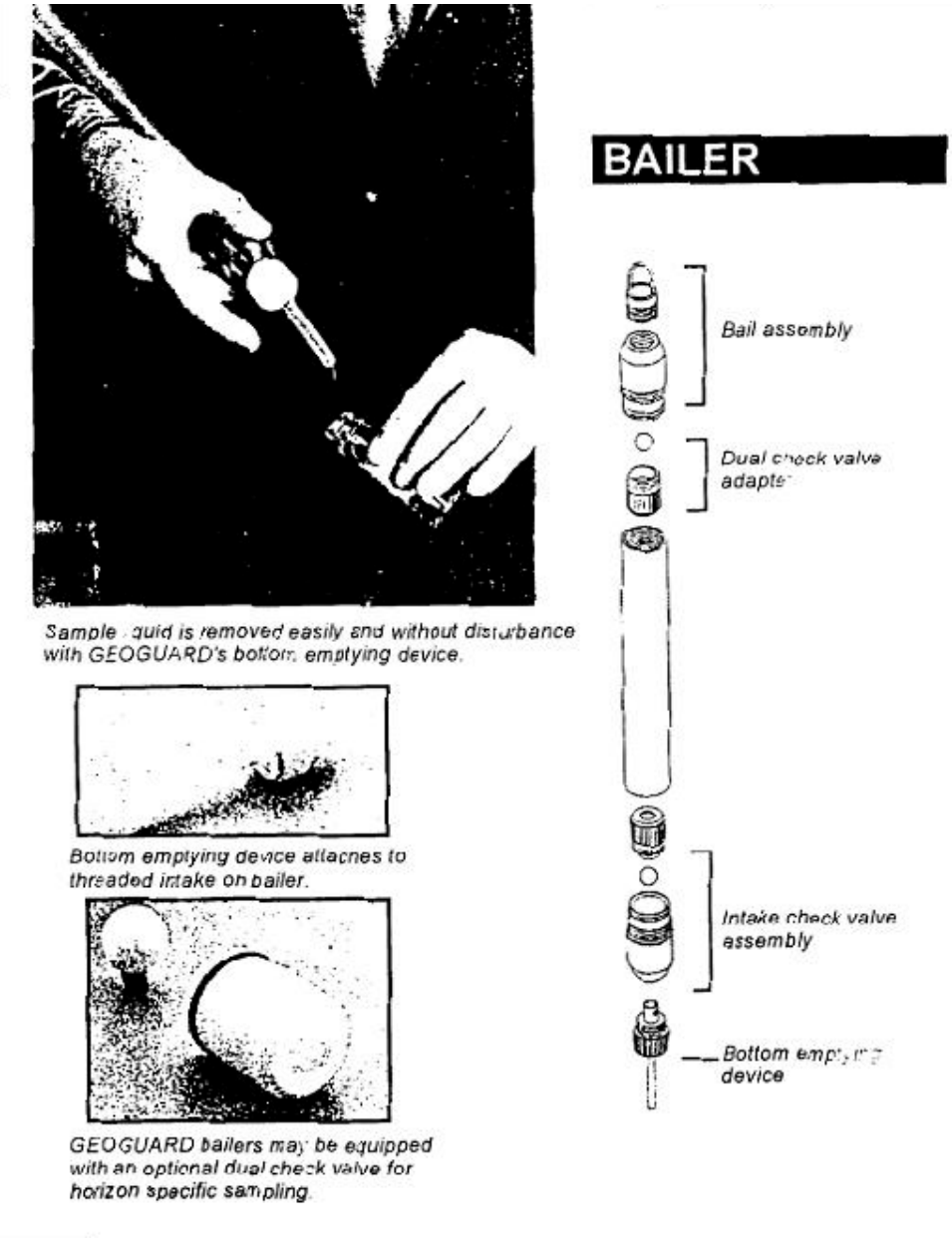
The following types of devices are examples of instruments used to collect environmental water samples:

- Bailers
- Dippers
- Collection directly into sample containers (Kemmerer sampler, Van Dorn sampler, grab sampler, lysimeter, weighted bottle samplers)
- Peristaltic pumps, bladder pumps, and other pump systems

Bailers

A bailer can be a cost-effective means of sampling when wells are shallow and slow to recover or when site access prohibits the transport of more elaborate systems. "Bailers are the oldest and simplest tools for sampling water wells. A bailer is a capped length of pipe attached to a rope, and is lowered and raised by hand, requiring no external power. . . " (*Procedures for the Collection and Preservation of Groundwater and Surface Water Samples and for the Installation of Monitoring Wells*, page 38). Over the years, bailer designs have been refined to permit more effective water sampling. For example, a dual check valve bailer is lowered through the water column with both valves open, allowing water to flow through. When the bailer is stopped at the desired horizon, both check valves close, trapping the sample between. The bailer is then retrieved and the sample removed through a bottom emptying device. See Figure 8-10, Bailer.

Figure 8-10. Bailer



Dippers

A dipper consists of a glass or plastic spoon attached to a pole, which serves as the handle. A dipper is used to sample liquids and can be typified by submerging a sample container. It may be used to recover water samples by immersing the holding container manually into the source and extracting the sample. The sample is collected and then transferred into a different bottle, which serves as the sample container. Stainless steel ladles and scoops, like kitchen utensils, may also be used as dippers. This type of collection results in greater aeration of the sample than collecting directly into the sample container.

Collection Directly into Containers

The sample container itself can be submerged in the water. The container opening should be pointing upstream, and the whole process should be conducted carefully, trying not to disturb the water. A weighted bottle sampler can be used to recover subsurface grab samples. Kemmerer and Van Dorn samplers are frequently used.

Peristaltic Pumps, Bladder Pumps, and Other Pump Systems

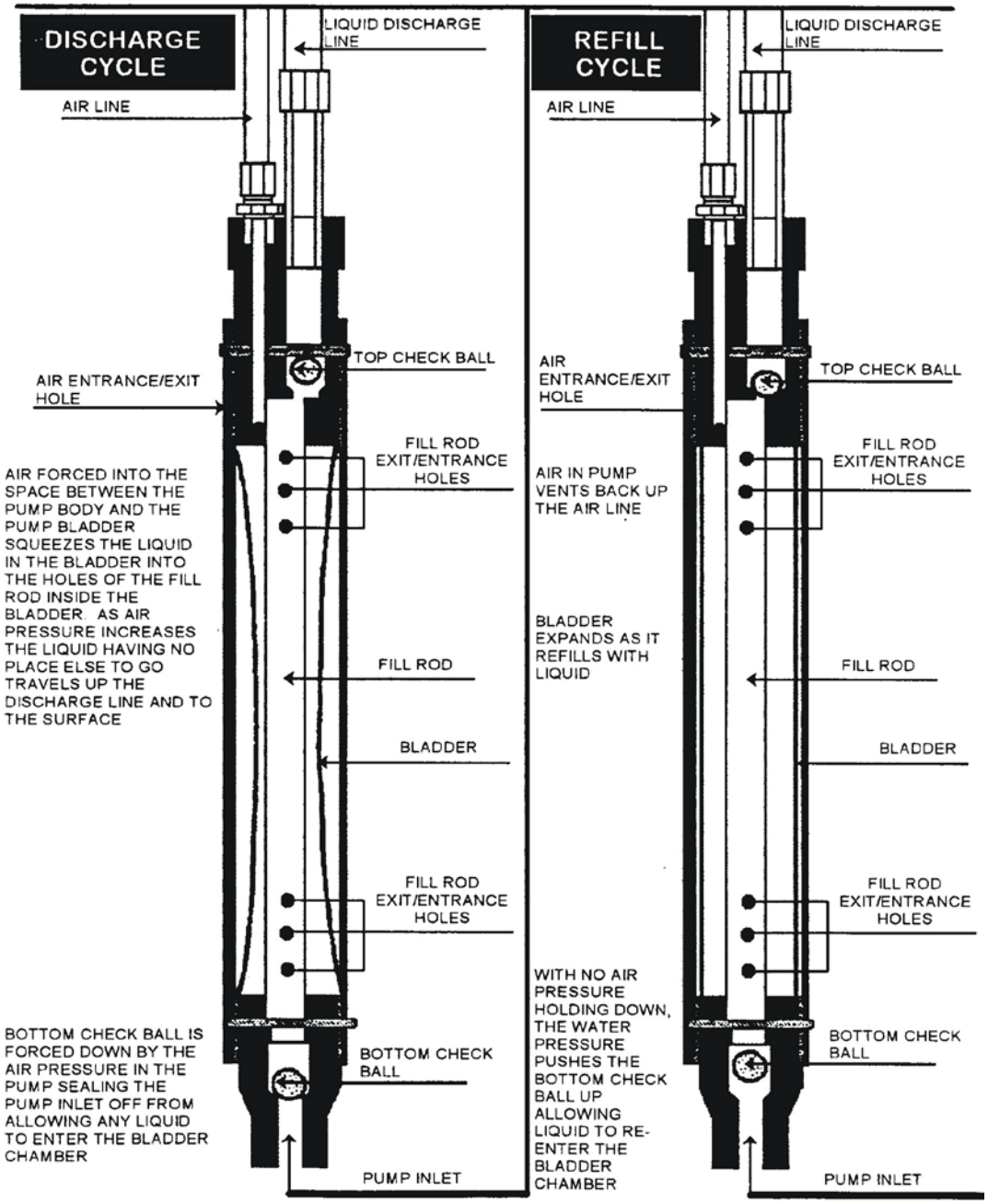
Peristaltic pumps are low-volume suction pumps that are suitable for sampling shallow, small-diameter wells. Their advantages include portability, versatility, relatively low cost, and limited risk of cross-contamination. A significant limitation is the degassing or stripping of volatiles that occurs in the sample, becoming most severe near the limit of suction lift. Use of this method is restricted to less than 20 feet.

Bladder pumps are also called gas-operated squeeze pumps. They use compressed air to pump water. Bladder pumps may be portable or installed for permanent use. Pump operation is described below:

- Water enters the pump through a check-valve on the bottom of the pump assembly.
- Compressed air inflates the bladder and forces the water to the surface.
- The compression release cycle is repeated until an adequate amount of water is obtained.

Bladder pumps have relatively slow pumping rates and require an air compressor for prolonged pumping because of their high rate of air consumption. They are generally used for groundwater sampling. See Figure 8-11, How the Bladder Pump Works.

Figure 8-11. How the Bladder Pump Works



Major Concerns of Water Sampling

Is the sample representative? The two major concerns in water sampling are the collection of a representative sample and the maintenance of the effluents/contaminants in their original concentrations before analysis. Because of this, it is important that the following considerations be addressed when sampling:

- Consideration of stream flow rate and variability will determine the number, location, and depth of samples taken. When sampling from continuously running sources, the effluent/contaminant is slowly being mixed with the water and other elements in the stream. It is important to determine the best method for recovering samples from the source.
- Homogeneity ensures that the sample medium is of essentially uniform composition, texture, appearance, etc. The major aims of quality control during sample preparation are to ensure that the sample is completely homogenized and no cross-contamination has occurred. Homogeneity is an important consideration in sampling and subsampling or sample splitting. If a material to be sampled is not homogeneous, multiple samples will be required in order to obtain a representative sample. Samples are homogenized so that measurements and subsamples are representative of the entire sample and thus the sampled area.

Self-Check Questions 8-4

INSTRUCTIONS: Fill in the missing words in each statement. Answers are located in the answer key section of the Trainee Guide. Choose from the following words.

appropriate	geography	maximum	release	thief
bailer	grab	mixing	representative	transfer
bypass	groundwater	overflow	residual	unrestricted
contamination	heights	Part 20 Appendix B	sample	upstream
continuous	holdup basin	pipet	shutdown	vapors
dipper	Kemmerer	pumps	surface	velocity
downstream	less	radionuclide	suspended	vertical
exposure pathway	low-volume		system	volume
				wells

1. Bottle sampling is suitable for extracting a _____ sample from storage tanks, tank trucks, or tank cars.

2. A _____sampler may be used to obtain samples from the bottom of tanks.

3. Tanks equipped for tap sampling generally have taps placed at several _____with a 1/4-inch pipe extending inside the tank shell.

4. Samples are removed from pipelines, filling lines, or transfer lines by passage through an area or medium and withdrawn into a sample container of_____size for analysis.

5. Sampling for on-stream analysis and control of process liquid or gas mixtures may be accomplished by using a sample cell and instrument installed in a _____of the main process pipe.

6. A special tap is installed in process lines so that a_____drip sample can be taken while process solutions are being transferred.

7. A_____ may be used to remove a sample from an open vessel. It may be attached to a stick for the purpose of extending the reach for withdrawing the material being sampled.

8. Tube sampling can be accomplished by using a _____ for the purpose of extracting a quantity of the material of interest.

9. Representative samples should be collected at each liquid release point for determination of the quantities and average concentrations of radionuclides discharged in liquid effluents that could reach _____ areas, including discharges to a sanitary sewage system.

10. For continuous releases, _____ samples should be continually collected at each release point.

11. The _____ is the pathway between the source of released material and intake by individuals.

12. Exposure pathway analyses for effluents and contaminants are impacted by _____ and demography.

13. All potentially contaminated liquid effluents generated in the site/facility (except mining and milling) are to be collected in a waste tank(s) prior to _____ to the environment.

14. Sampling and analyzing waste tank contents provides assurance that concentrations at the point of release are below the NRC 10 CFR _____ concentration limits and that water quality meets the state requirements and those of the EPA water quality standards and criteria.

15. Water from building roof drains, yard drains, and storm water runoff should flow to a _____, where samples are taken following precipitation and analyzed to ensure that any low-level contaminants are below the regulatory limits.

16. Sewage sludge should be sampled for possible _____ accumulation.

17. The _____ of the liquid effluents should be reported so NRC staff can calculate the quantity of radionuclides discharged.

18. A glass or plastic jar with a screw cap may be used to obtain a _____ in a free-flowing stream.

19. Collecting discrete samples at various depths in shallow water can be accomplished by using a Niskin or _____ sampler.

20. When sampling _____ solids, it is important to sample the upper and middle portions of a tank separately from the bottom sample (foots).

21. Multiphase liquids may also require _____ prior to sampling.

22. Many liquids give off _____ that are toxic or flammable, so adequate ventilation and care against ignition are necessary.

23. Slurry _____ is the movement of a liquid in which solid or semisolid materials are suspended.

24. Although different materials exhibit different settling characteristics, settling is most likely to occur at _____ or when flow rates are reduced because of abnormal operations.

25. Visual access should be provided to all surfaces or spaces where material is likely to accumulate; alternatively, clearance should be provided to permit external use of nondestructive assay instruments or internal probes to detect the presence of special nuclear material or to identify the location of _____ material not visually accessible.

26. Effluent _____ and corrosion can significantly affect in-line sampling and measurement.

27. Components of the sampling _____ should be accessible for maintenance.

28. The most important aspect of an environmental water monitoring program is the detection of _____.

29. _____ sampling is conducted to provide information about the condition of subsurface water resources in the zone of saturation.

30. Groundwater samples are usually obtained from _____ through the use of a variety of pumping and filtering devices.

31. Drinking water may be supplied from _____ water or groundwater.

32. Surface water drinking sources should be sampled from treated water at the point of _____ concentration of contamination or release and from untreated water.

33. The best locations to sample in rivers and streams are _____ of turbulence where contaminants are more likely to be well mixed.

34. Background samples are typically taken _____ of the facility discharge.

35. It is often recommended that _____ composites be collected for a sample at a given position along a stream or river.

36. Lakes and ponds experience _____ mixing and have a greater tendency to stratify than streams and rivers.

37. In relation to storm runoff, it is important that the potential for _____ be addressed and monitored to ascertain whether a significant change in pathway has been created.

38. A _____ can be a cost-effective means of sampling when wells are shallow and slow to recover or when site access prohibits the transport of more elaborate systems.

39. Peristaltic pumps are _____ suction pumps that are suitable for sampling shallow, small-diameter wells. Use of this method is restricted to less than 20 feet.

40. Bladder _____ are generally used for groundwater sampling.

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

8.1.7 Identify techniques used in sampling gases.

SAMPLING GASES

Evaluating the impact of airborne contaminants necessitates the accurate determination of the concentration of the contaminant present in a unit volume of air. The value is determined either directly from an airstream or, following collection of the contaminant, from a known volume of air on a suitable medium. The analytical methods for analysis of the material will depend upon the material's physical state (i.e., gas, vapor, or particle) and upon its chemical composition.

Significance of Airborne Pathway

The airborne pathway is particularly significant because it comprises both gases and particulate matter and is interdependent and closely associated with meteorology and the atmosphere. For example, some contaminants in fuel cycle facilities have adverse reactions when released to the air (e.g., uranium hexafluoride). These contaminants can be dispersed into the air and transported by the wind.

Exposure can be through inhalation or contact or by ingestion. The fact that airborne effluents and discharges can be ingested after deposition on foodstuffs is particularly significant to the exposure pathway.

Air Sampling Techniques

Three air sampling techniques are:

- Air filtration
- Absorption and adsorption
- Impaction and impingers

Air Filtration

Air filtration is the most common technique used to collect particulates. Air particulate samples are normally collected on a filter medium with an air pump and a flow-measuring device. Samples can be used individually or composited. The air sampling system must have a flow-rate or flow-integrating meter that is mounted in an all-weather shelter, with the sampler discharge located so as to prevent the recirculation of air. The collection of particles by air filters is achieved through impaction, diffusion, interception, and electrostatic attraction. Air-moving systems for gaseous effluent sampling should maintain a constant airflow during sampling conditions.

Adsorption

Adsorption is the most commonly used method for collecting gases and vapors. Adsorbers are solid granules with large surface areas. They are packed into cartridges or tubes. Gaseous contaminants are collected when they adhere to the solid bodies of the surfaces they contact as they pass through those cartridges or tubes. Except during sampling, the adsorber should be completely sealed to prevent passive adsorption of unwanted material or loss of the desired material that has been collected. Analysis should be performed as soon after sampling as possible. Two of the most common adsorbers are listed below:

- Activated charcoal, which is used to collect radioiodine and noble gases
- Silica gel, which is generally used to collect water vapor but is also used for collection of alcohol, aldehydes, ketones, esters, and aromatics

Absorption

Absorption is performed by liquids inside devices called bubblers or impingers. The airstream entering the device is bubbled through the solution and then drawn off. A solution is chosen for use that will dissolve the gas or vapors of interest. For example, a hydrogen peroxide solution can be used to dissolve (i.e., collect) sulfur dioxide. Glass bubblers are usually preferred, since they are less prone to leaks. While there are many types of absorber solutions, these are the three most common:

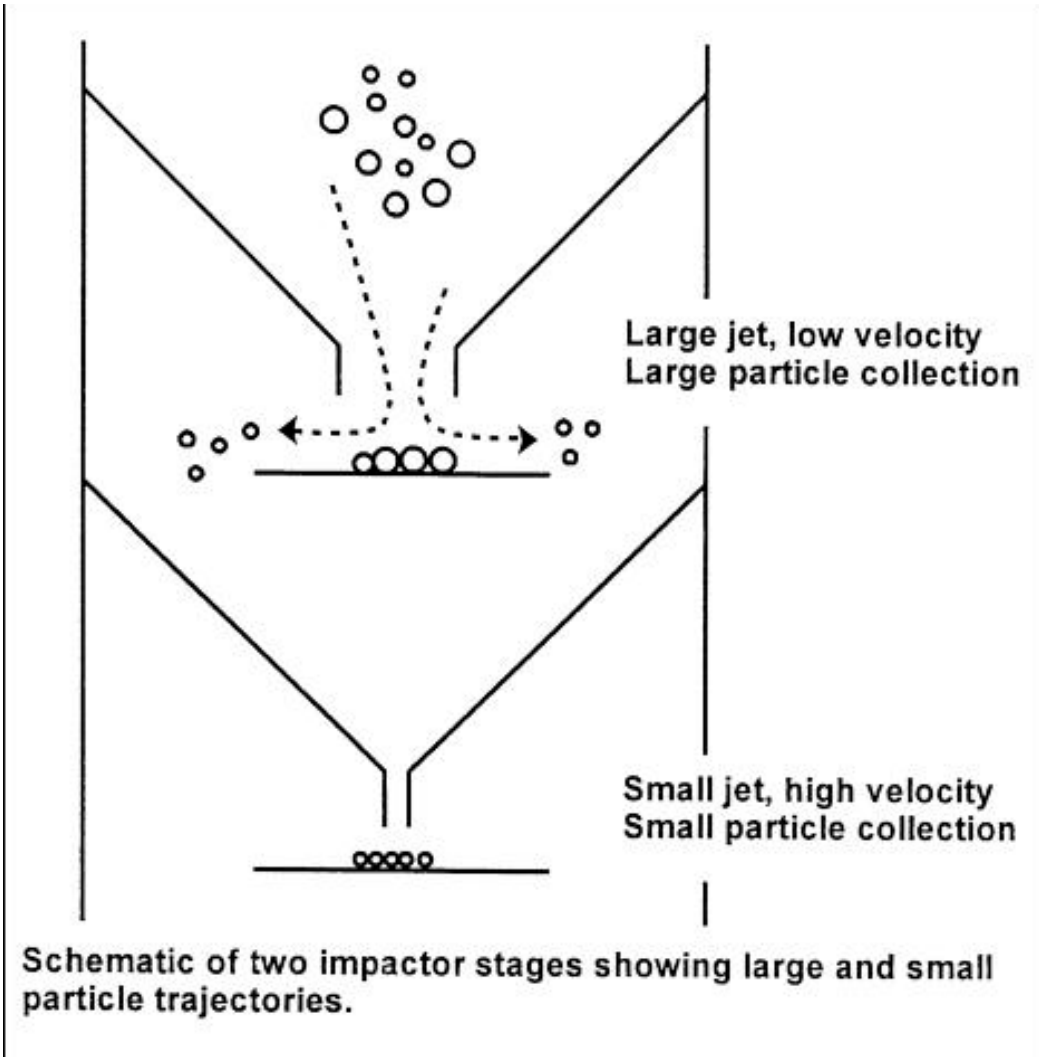
- Ethylene glycol is used to collect tritiated water vapor. For analysis, aliquots of the solution are transferred to liquid scintillation cocktail and counted.
- Hydrogen peroxide (H_2O_2) is the standard method used to collect sulfur dioxide.
- Sodium hydroxide (NaOH) is the most common method used to collect carbon dioxide.

To avoid errors in determinations of the volumes of air sampled, it is extremely important that careful attention be paid to the use and calibration of the flowmeter. A suitable pump should be as trouble-free as possible. It is essential to employ an elapsed timer that indicates how long the pump has been running. A flow regulator on the pump assists in maintaining a constant flow during filter loading.

Particle Impaction

Particle impaction is the collection of particles that deviate from the airflow stream. Impaction occurs when the stream bends as the airflow bypasses a solid object. Particles become imbedded on the solid surface that they impact. See Figure 8-12, Particle Impaction.

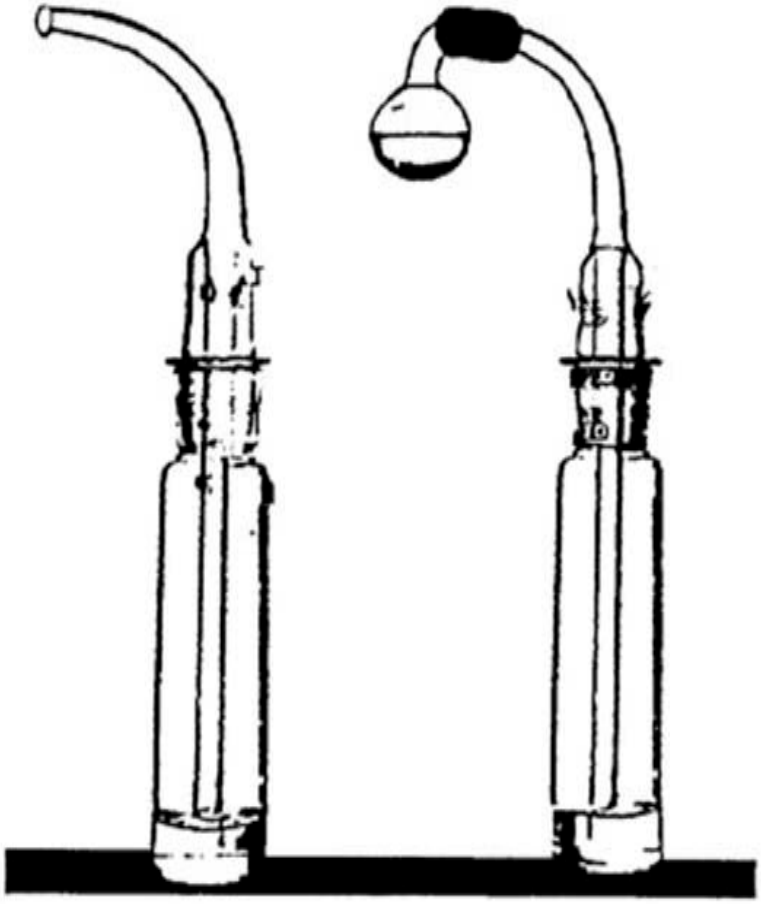
Figure 8-12. Particle Impaction



Impingers

Another method of collecting airborne particulates is by use of an impinger. A glass flask absorber receives air at a relatively high velocity through a small glass nozzle. The nozzle directs the air so that it strikes the bottom of the flask, which is partially filled with a liquid, such as water or alcohol. The dust particles are wetted and retained in the liquid. The liquid can then be analyzed to detect the presence of contaminants of concern in the atmosphere. See Figure 8-13, Glass Impingers Used for the Sampling of Viable Particles. Impingers operate much like impactors.

Figure 8-13. Glass Impingers Used for the Sampling of Viable Particles



SAMPLING EQUIPMENT

In general, the type of equipment typically needed to sample gases consists of a sampling probe, a sample delivery line, and a container to receive the sample. The sampling probe is that part of the sampling line attached to and extending into a pipe or vessel. The connections to the vessel may be threaded or welded pipe fittings. Sampling lines may be pipe or flexible tubing of a suitable resistant material. Gas purging, water displacement, or vacuum purging may be the method used to fill the vessel with the gas to be sampled. The container may be used simply as a means of conveying the sample from the source to the analytical apparatus, or it may contain an absorbent for collection of specified constituents of the gas. Minute quantities of materials can be extracted from the air for trace contaminant analysis with specially designed syringe samplers. Such devices can be used to withdraw microliter samples from the source and transfer them to a measuring instrument. Syringe samplers can be equipped with tubes that contain absorbent materials that are subsequently analyzed for trace contaminants.

Sampling Devices

Air sampling devices are listed below:

- High-volume filters
- Charcoal canisters
- Detection indicators
- Detectors for real-time analysis

High-Volume Filters

High-volume filters are used in flow rate metering instruments for collection of airborne effluents. It is important to know the actual clock time at the start and stop and the elapsed time when conducting air sampling with high-volume air filters.

Environmental air sampling must account for dispersal as well as lower acceptable limits. High volume samples are generally taken to offset very low detection limits.

During air sampling, volume is important. It is important that sampling devices be calibrated and checked regularly. Results may fluctuate widely due to meteorology. Temperature inversions cause airborne materials to concentrate near the earth. High winds may cause dispersal and thus lower concentrations but may also pick material up and cause higher airborne levels. Precipitation generally removes contaminants from the air. One common form of interference is natural radon. Its decay products, which are particulates, collect on filter media and provide additional alpha and beta activity. This can be minimized by allowing the radon to decay prior to measuring, which takes about six hours. Thus, for environmental air samples, it may be necessary to wait at least four hours before counting samples.

Charcoal Canisters

Charcoal canisters can be used in a manner that resembles tube sampling for air contaminants. The charcoal traps gases present in the air. Charcoal canisters may also be used in conjunction with another filter. The filter removes particulates from the air, while the charcoal traps gases. Activated charcoal, which is used in charcoal canisters, is primarily used to collect radioiodine. It will also collect noble gases, but its efficiency for these is relatively low. Charcoal is also excellent at collecting and retaining many vapors.

Detection Indicators

Three types of direct-reading colorimetric indicators have been used for the determination of contaminant concentrations in air:

- Liquid reagents
- Chemically treated papers
- Glass indicating tubes containing solid chemicals

Detectors for Real-Time Analysis

Detectors for real-time analysis may operate in a semicontinuous manner by filtering airborne particles onto a portion of continuous tape for a specified time period. The sample is then counted by a detector just above or beneath the tape, after which the tape moves to provide a clean substrate for the next sample.

An example of a detector, used to monitor rather than sample radioactive isotopes of noble gases is an environmental particulate iodine noble gas monitor (PING). In this system air is passed through a filter to remove the particulates; charcoal to remove the iodines; and a flow-through detector, which responds to the only radioactive material left in the airstream, the noble gases. The response of the flow-through chamber must be calibrated with a mixture of the gases known to be released from the facility.

SAMPLING AIRBORNE EFFLUENTS

Airborne effluents should be continually sampled unless the license has established that radioactivity in the effluent is insignificant and periodic sampling is sufficient. If periodic sampling is used, the samples should be representative of actual releases.

According to U.S. NRC Regulatory Guide 8.37 ALARA Levels for Effluents from Material Facilities, 10 CFR 20, "Standards for Protection Against Radiation," states that a "licensee shall show compliance with the annual dose limit in Part 20.1301 by (1) Demonstrating by measurement or calculation that the total effective dose equivalent to the individual likely to receive the highest dose from the licensed operation does not exceed the annual limit; or (2) Demonstrating that (I) the annual average concentrations of radioactive material released in gaseous and liquid effluents at the boundary of the unrestricted area do not exceed the values specified in Table 2 of Appendix B to Sections 20.1001-20.2401; and (ii) if an individual were continually present in an unrestricted area, the dose from external sources would not exceed 0.002 rem (0.02 mSv) in an hour and 0.05 rem (0.5 MSv) in a year."

The sampling rate at each release point should be such that a representative sample of the effluent is collected. The volumes of gaseous effluents should be reported so NRC staff can calculate quantities of radionuclide discharge.
A preoperational assessment should be made and documented in "The Applicant's Environmental Report" to determine the types and quantities of airborne effluents to be expected from the facility and to establish the associated airborne effluent monitoring needs of the facility.

Point and Diffuse Sources

The sources contributing to the total emissions from a facility can be considered as either "point" or "diffuse" sources. A **point source** is a single defined point of origin of an airborne release, such as a stack. A **diffuse source** is an area source or several sources of radioactive contaminants released into the atmosphere (all sources other than point sources). Diffuse sources are difficult to characterize. They should be identified and assessed for their potential contribution to public dose. Examples of diffuse sources are ponds, contaminated areas, structures without ventilation, or ventilation that does not result in a well-defined release point.

STACK SAMPLING

Purposes

Airborne effluents in a stack consist of gases, vapors, and/or particulates. The purposes of stack sampling are to do the following:

- Determine atmospheric concentrations of radionuclides in order to allow an estimation of doses to the public
- Indicate short-term excessive concentrations requiring corrective measures
- Indicate long-term variations in releases that may indicate deteriorating equipment
- Evaluate new procedures
- Assist in materials balancing
- Demonstrate compliance with regulatory requirements

Once well mixed, the distribution of gases and vapors across an airstream in a stack is unlikely to change. This distribution is not affected by gravity, nor is it affected when the airstream changes direction. The only thing that might affect the distribution would be the introduction of another airstream.

On the other hand, the distribution of particulates across the airstream in a stack or duct can be affected by gravity and can change as the air changes direction. These changes primarily involve the large particulates; small particulates (i.e., those with aerodynamic diameters less than one micron) tend to behave like gases. Some of the larger particulates can become completely lost from the airstream.

MODULE 8.0: SAMPLING AND MEASUREMENT PRACTICES

Goal

The goal of stack sampling is to permit a calculation of the emission rate from the stacks of a fuel cycle facility in Ci/s or other appropriate units. By determining the average velocity of the effluents and measuring the concentration of the contaminant in an extracted sample representative of the effluents, the site/facility can characterize and quantify the effluents being discharged.

Sampling the Effluents

Since it is impossible to analyze the entire effluent, small portions (samples) are collected and analyzed. It is extremely important that the sample be representative of the effluent as a whole.

The term continuous monitoring refers to the process by which a real-time (instantaneous) analysis of the stack effluents is performed. An on-line monitoring system might employ a Geiger-Müeller detector placed inside, or adjacent to, the stack. An off-line monitor requires that a portion of the effluent be extracted from the stack and delivered to the monitor for analysis. PINGs are examples of off-line monitors.

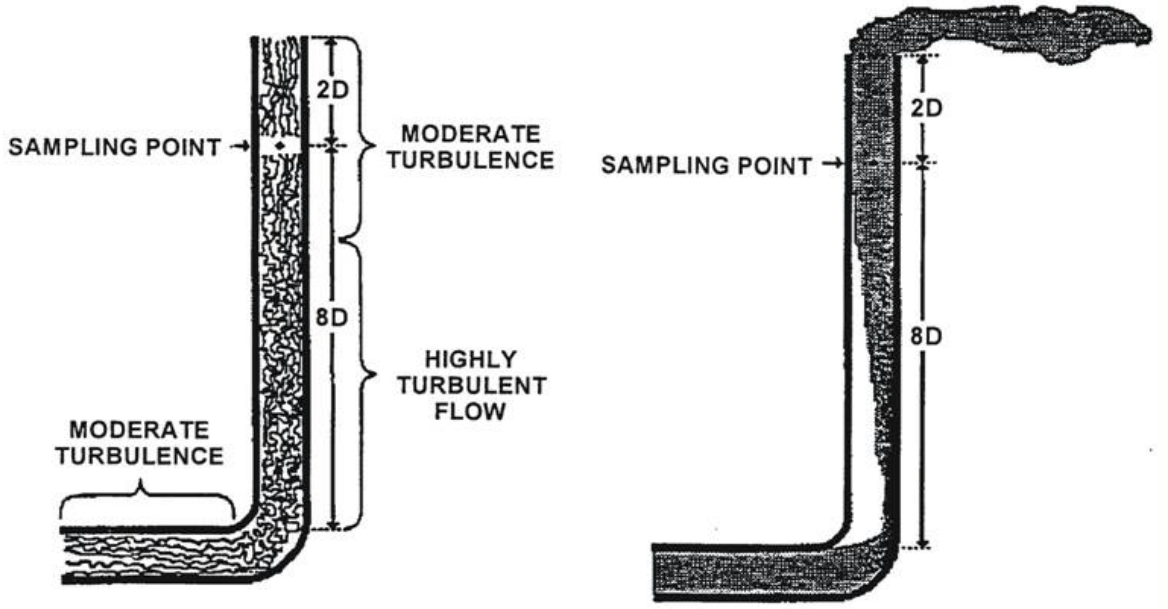
Stack Sampling Location

Since the purpose of stack sampling is to evaluate what is being released into the atmosphere, the sampling location should be downstream of the filter and as close to the point of release as possible. The sampling location should also be away from areas of the stack where the flow is being disturbed (i.e., filter housings, bends, blowers, cyclones, constrictions, and expansions). The sampling point should be at least eight stack-diameters downstream and two diameters upstream from any disturbance to the stack airflow.

Another requirement for stack sampling is that the contaminants be evenly mixed across the effluent. This necessitates that sampling be performed well downstream of points in the ventilation system where two ducts converge, a place where the gaseous component of the effluents as well as the particulates might not be evenly distributed. When possible, the sample should be extracted from a vertical stack rather than a horizontal stack, where the particulates may settle due to gravity and concentrate toward the bottom of the effluent stream.

Sampling eight diameters downstream and two diameters upstream from disturbances to the airflow also improves the chance that the particulates will be thoroughly mixed. Figure 8-14, Turbulence Inside the Stacks and Appropriate Sampling Height, shows how the air entering the bend at the base of the stack becomes highly turbulent. The drawing on the right shows how the particulates entering the bend are carried toward the far side of the stack by their own momentum.

Figure 8-14. Turbulence Inside the Stacks and Appropriate Sampling Height



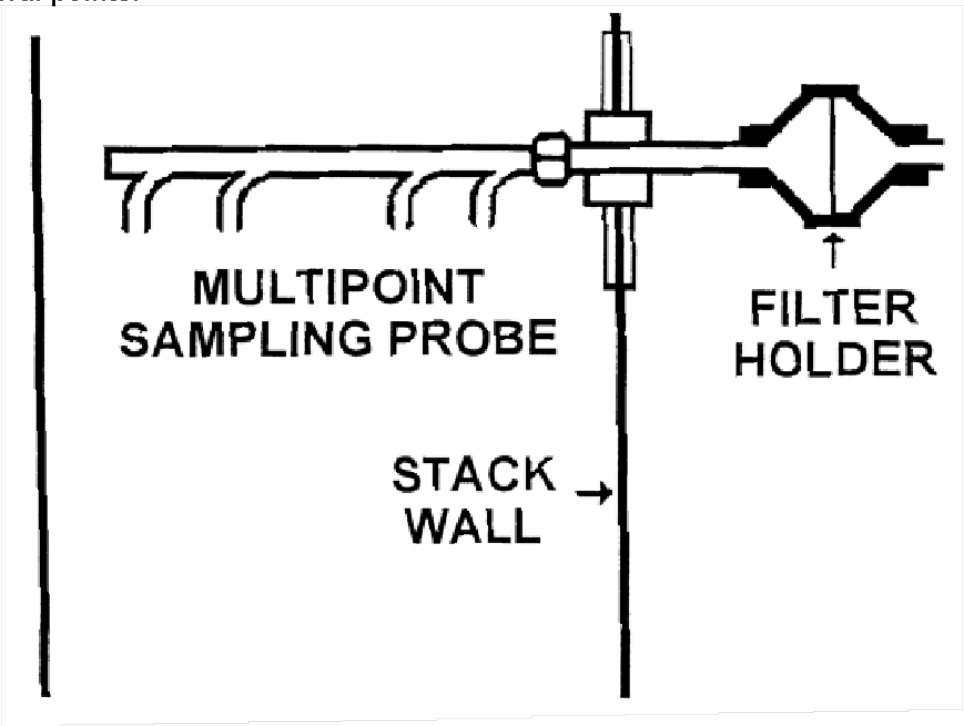
Sampling here would be inappropriate because of the highly turbulent flow and the uneven distribution of particulates. The turbulence at the bend mixes the effluent so that, by eight diameters downstream, the particulates are evenly distributed again. The even distribution of the particulates, together with the fact that the turbulence has settled down, makes this an excellent sampling location.

Nozzles

The withdrawal point(s) that permits sampling is called a nozzle. It is rare to find more than one nozzle in a stack that is less than 24 inches in diameter. Tests are conducted that determine the appropriate number and placement of nozzles for stack sampling. Figure 8-15, Multipoint Sampling Probe, illustrates a type of probe (nozzle assembly) that permits simultaneous sampling at several points.

Figure 8-15. Multipoint Sampling Probe

This figure illustrates the type of probe/assembly that will permit simultaneous sampling at several points.



Nozzle Location Inside the Stack

The velocity of the effluent at the sampling point should be the same as the average velocity across the stack. The assumption is that the average concentration of the contaminants will also be found at this point. The centroid, shown in Figure 8-16, Centroid Location, is the line that divides the cross-sectional area of a round stack into an inner circle and an outer annulus of equal areas. Studies indicate that a representative sample can usually be obtained eight stack-diameters downstream if the sampling point inside the stack is located at a distance between one-tenth and three-tenths of a diameter from the stack wall. The centroid is located in this range.

Figure 8-16. Centroid Location

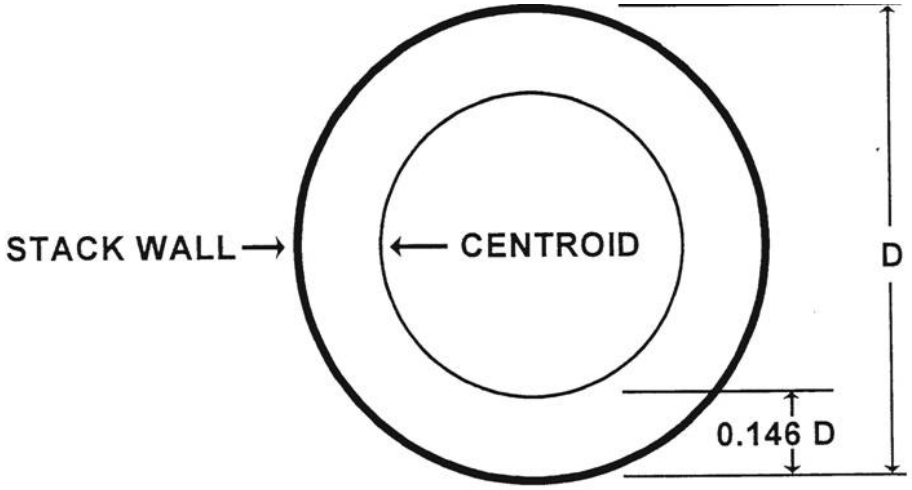
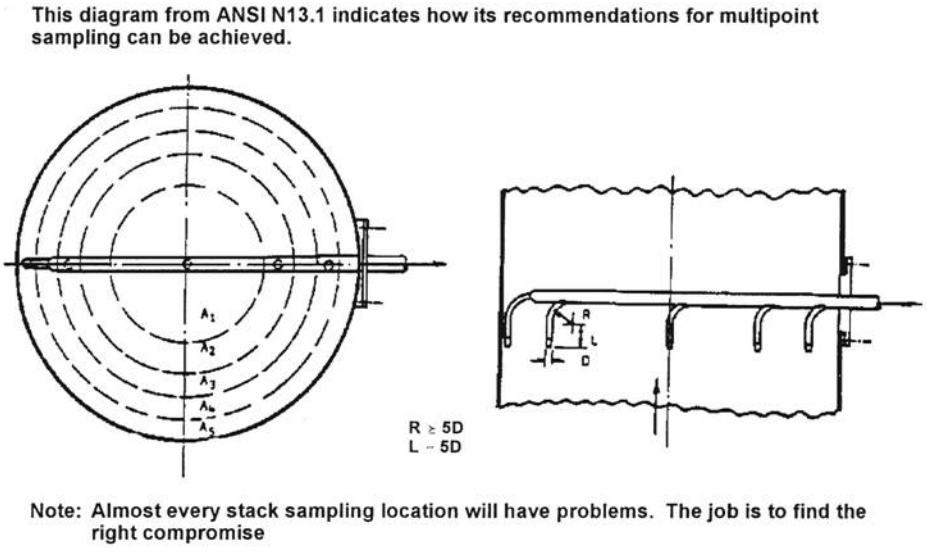


Figure 8-17, Recommendations for Multipoint Sampling, from American National Standards Institute (ANSI) N13.1 illustrates recommendations for multipoint sampling.

Figure 8-17. Recommendations for Multipoint Sampling



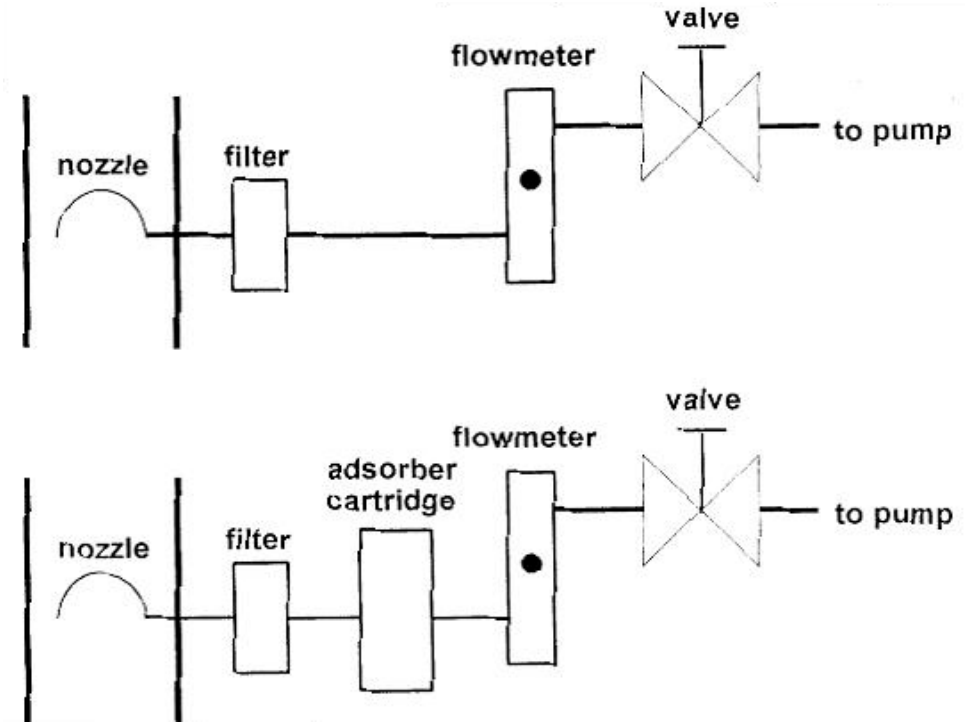
Sampling Trains

The term sampling train refers to the sequential arrangement of sampling equipment. A normal sequence for a sampling train is listed below:

- Nozzle
- Collection medium for particulates
- Collection medium for gases and vapors
- Flowmeter
- Pump

In a typical stack sampling train, the collection medium used for particulates is almost always a filter. The collection media used to collect the gaseous components of the effluent include adsorbers, absorbers, cold traps, etc. All sampling trains will sample for particulates and at the very least consist of a filter, flowmeter, and pump. See Figure 8-18, Examples of Stack Sampling Trains.

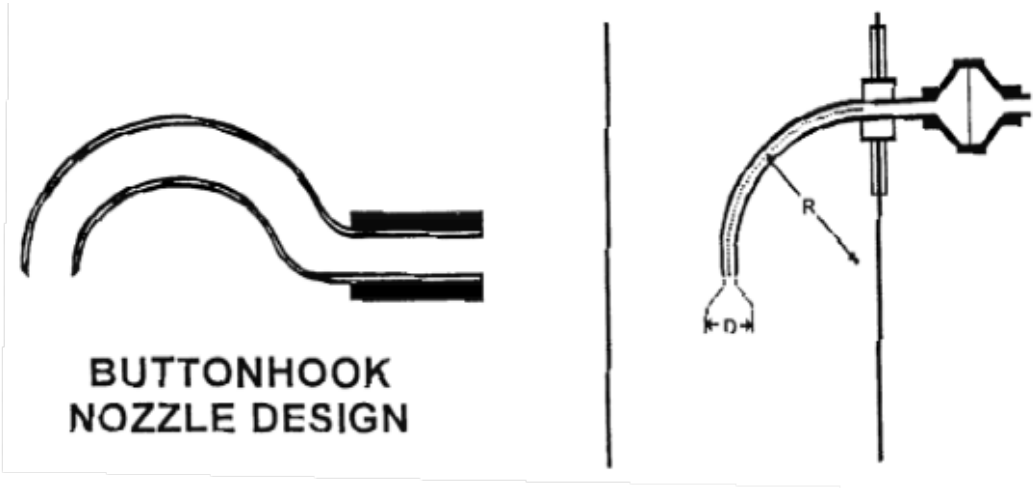
Figure 8-18. Examples of Stack Sampling Trains



Nozzle Design and Care

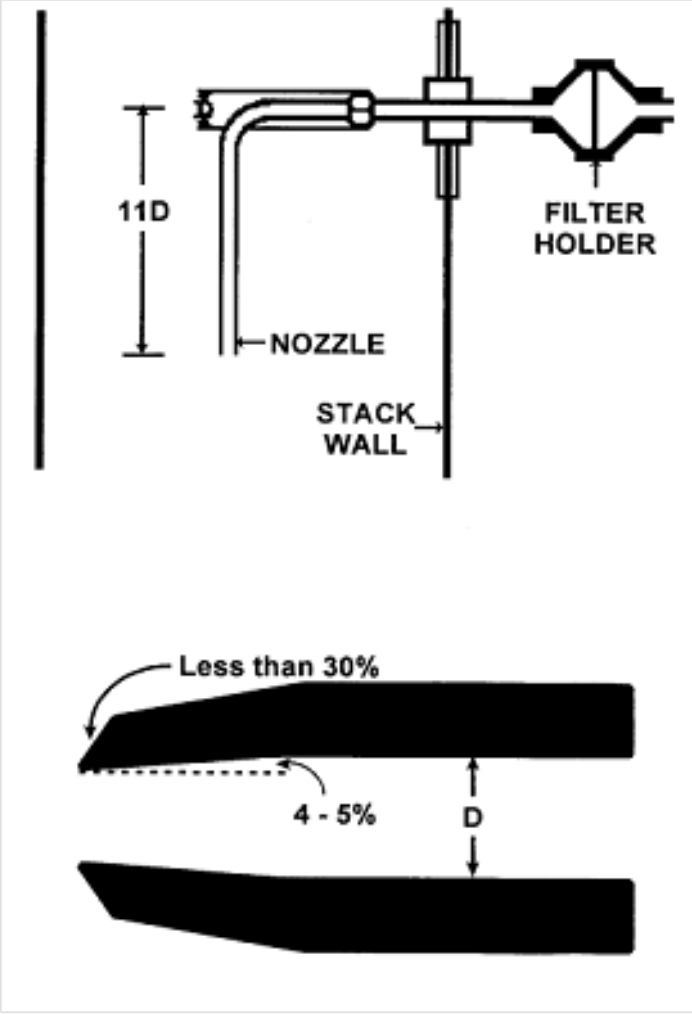
Nozzles are available in various shapes and sizes. Probe and nozzle materials should be noncorrosive, easy to clean, and rigid. While stainless steel is usually the material of choice, other materials, such as glass and even Teflon, have been used. In general, larger nozzle openings are preferred because they are less likely to become plugged. Figure 8-19, Common Nozzle Designs, illustrates two common nozzle designs. The figure on the left is called a buttonhook nozzle, and the nozzle on the right is a simple, smooth, curved nozzle.

Figure 8-19. Common Nozzle Designs



Because turbulence created by the nozzle and the stem of the probe can impair the collection of a representative sample, the design of the nozzle and its supporting assembly should be streamlined and no bulkier than is absolutely necessary. Figure 8-20, Nozzle Design, illustrates nozzle placement inside the stack.

Figure 8-20. Nozzle Design



Nozzles should be inspected, sharpened, cleaned, realigned, or replaced periodically. Besides general maintenance, it is also important to observe appropriate safety precautions when working around stacks. For example, when an individual is inserting a probe into the stack, the effluents (which may be toxic in some cases and will be under positive pressure) can blow directly into his or her face.

Filters

The particulate component of the stack effluent is almost always collected on a filter. Filters are available in a variety of sizes and types.

The filter should be as close to the source as possible in order to reduce the loss of large particulates in the sampling line upstream of the filter. Inevitably, some particles will collect in the nozzle and the line. The ideal solution is to recover these by cleaning out the nozzle and line during each filter change. The more practical solution is to assume a long-term equilibrium situation in which the losses are balanced by particles reentering the air stream.

Types of Filters

The type of filter chosen depends on loading expected, physical strength required, and type of analysis to be performed. There are three major categories of filters:

- Cellulose
- Glass fiber
- Membrane

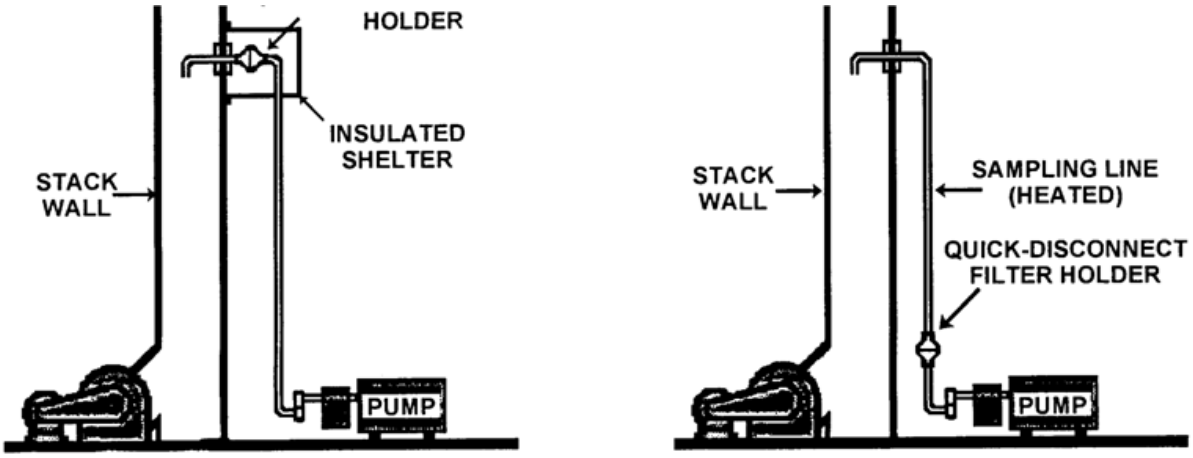
Table 8-3 provides the advantages and disadvantages of the three types of filters.

Filter holders are required for placement of the filters in the sampling train. They are often a site for leaks and should be inspected and cleaned on a regular basis. To simplify sample collection, filter holders with a quick-disconnect coupling can be used. These couplings should be made of stainless steel or brass. See Figure 8-21, Filter Holders.

Table 8-3. Advantages and Disadvantages of Cellulose, Glass, and Membrane Filters

Type of Filter	Advantages	Disadvantages
Cellulose	◙ Cellulose filters are inexpensive. ◙ They are mechanically strong. ◙ They are readily decomposed by acid to permit radiochemical analysis of pure beta and alpha emitters.	◙ Efficiency is affected by the velocity of air across the filter (the higher the velocity, the higher the efficiency); as the filter accumulates particulates, there is an accompanying increase in collection efficiency, which introduces a small uncertainty into the estimate of the efficiency. ◙ They will suffer burial losses if alpha particles are being counted. ◙ They are hygroscopic.
Glass fiber	◙ The collection efficiency of glass filters approaches 100%. ◙ The efficiency is not affected by sampling flow rate (i.e., they have a low blockage rate). ◙ Burial losses of alpha emitters can be low to nonexistent.	◙ They are highly resistant to chemical decomposition. ◙ Glass-fiber filters are not easily digested for radiochemical analysis, except using hydrofluoric acid.
Membrane	◙ Burial losses for alpha emitters are nonexistent. ◙ The collection efficiency is 100% no matter what the flow rate. ◙ Membrane filters are easily digested for radiochemical analysis.	◙ They are highly resistant to airflow (high pressure drops). ◙ Resistance to airflow greatly increases as the filter loads (i.e., have a high blocking rate). ◙ They are expensive. ◙ They are fragile.

Figure 8-21. Filter Holders



Sampling Lines

Sampling lines should be made of materials that will not react chemically with the effluents in the airstream. It is also important that the insides of the lines are smooth and that none of the system components upstream of the filter create a static charge that could potentially remove particulates from the airstream. Stainless steel is the best choice for sampling lines.

Normally the particles in the stacks will be small. Particles smaller than one or two microns tend to behave like gases and show little tendency to settle in sampling lines.

Leaks in Sampling Lines

The system should be checked periodically for leaks. Areas where leaks are likely to arise include the following:

- Tubing connections
- Filter or cartridge holders
- Bubblers/impingers

Alarms

To signal the need for corrective actions that may be necessary to prevent public or environmental exposures from exceeding established limits when continuous monitoring systems are required, alarms should be used that provide a warning.

Gaseous effluents are predominantly from point sources and are treated to control airborne radionuclides to near-background levels. The cumulative effect of low-level releases may result in reaching the criteria for continuous emission monitoring. Environmental sampling and measurement provide an additional level of measurement so these releases are detected.

EXAMPLE OF FORMAT FOR REPORTING EFFLUENT DATA

This is for illustration only and is not a complete listing of data to be reported. Supplemental and explanatory information should also be submitted. Significant systematic errors should be reported, if appropriate, in supplemental information. Calculation of the lower limit of detection (LLD) should be shown in supplemental information also.

1. CONTINUOUSLY SAMPLED STACKS

For each release point, report the following information:

- Reporting period
- Stack location (process or area)
- Stack flow rate (m3/sec) or total stack flow (m3) (if stack is not in continuous use)
- Radioactivity (Ci)
  - Gross alpha
  - Gross beta

Radionuclide	Concentration (μ Ci/ml)	Error Estimates (+ μ Ci/ml)	LLD (μ Ci/ml)	Quantity Released (Ci)	Derived Air Concentration (DAC)

2. OTHER SAMPLED STACKS

For each release point, report the following information:

- Date(s) sampled
- Stack location
- Total stack flow (m3)
- Radioactivity (Ci)
  - Gross alpha
  - Gross beta

Radionuclide	Concentration (μ Ci/ml)	Error Estimates (+ μ Ci/ml)	LLD (μ Ci/ml)	Quantity Released (Ci)	Derived Air Concentration (DAC)

ANALYSIS OF GASEOUS SAMPLES

The type and frequency of analysis depend on the requirements established by the site/facility. For example, at nuclear fuel processing and fabrication operations, 10 CFR 70 requires that gaseous samples be analyzed at least weekly for gross alpha and beta activity. For uranium hexafluoride production (licensed under 10 CFR 40), gaseous samples should be analyzed at least weekly and liquid samples at least monthly for gross alpha activity.

Radionuclide analysis should be performed on selected samples unless (1) the gross alpha and gross beta activities are so low that individual radionuclides could not be present in concentrations greater than 10% of the quantities specified in Appendix B of 10 CFR 20 or (2) the radionuclide composition of the sample is known through such operational data as the composition of the feed material.

The following examples are cases in which operational data may not be adequate for the determination of radionuclide composition:

- Plants processing uranium in which extraction, ammonium diuranate precipitation, ion exchange, or other separation processes could result in concentration of thorium isotopes (principally thorium-234)
- Plants in which uranium of varying enrichments is processed during the period of consideration
- Plants processing plutonium in which significant variation in the plutonium-238/plutonium-239 ratio among batches of plutonium and the continuous in-growth of americium-241 would preclude the use of feed material data in determining the radionuclide composition of effluents
- Uranium hexafluoride production plants in which evaluations based on feed materials show significant changes in the radionuclide ratio (e.g., uranium, radium, thorium)

Radionuclide analyses should be more frequent under the following conditions:

- At the beginning of the monitoring program until a predictable radionuclide composition of effluents is established
- When there is a significant unexplained increase in gross radioactivity
- When a process change or other circumstance might cause a significant variation in the radionuclide composition

The results of analyses of the samples for each release point should be used to determine the following:

- The total gross alpha and gross beta activity (as applicable) discharged
- The average concentration of gross alpha and gross beta activity (as applicable) discharged
- The total activity and average concentrations of each of the radionuclides discharged

Lower Limit of Detection

The LLD is defined as the smallest concentration of radioactive material sampled that has a 95% probability of being detected. (Radioactive material is "detected" if it yields an instrument response that leads the analyst to conclude that activity above the system background is present.)

The lower limit of detection for any analysis should not be more than 5% of the concentration limits listed in Appendix B of 10 CFR 20.

If the concentrations of radionuclides being sampled are known to be higher than the lower limits of detection indicated, the sampling and analysis procedures need only be adequate to measure the actual concentrations. The LLD should be low enough to accommodate fluctuations in the concentrations of the effluent and the uncertainty of the LLD.

Reporting Results

All sampling and measurement results data should be summarized in report format on a semiannual basis. For each release point, the following information should be reported as appropriate:

- Type of sample
- Sampling location
- Dates during which samples were collected
- Quantities of gross alpha activity, gross beta activity, and each radionuclide released

- Average concentrations of gross alpha activity, gross beta activity, and each radionuclide released
- Percentages of the appropriate concentrations listed in Appendix B of 10 CFR 20

The following summary should be included in each report:

- For all gaseous releases, the total quantities of gross alpha activity, gross beta activity (if appropriate), and each radionuclide
- For the gaseous sample observed to contain the highest concentrations of radioactivity for the semiannual period, the concentrations of gross alpha activity, gross beta activity (if appropriate), and each radionuclide, along with the percentages of the appropriate concentrations listed in Appendix B of 10 CFR Part 20

If the highest concentrations are less than 10% of those listed, the summary should indicate that the result was below the stated appropriate value.

Reported results should include error estimates. The standard deviation representing the random error of the analysis should be reported for each result. An estimate of the magnitude of the systematic error should be reported separately. Results reported as below the lower limit of detection need not include error estimates. The LLD should be included.

Supplemental Information for Reports

The following supplemental information should be included in the first effluent monitoring report (subsequent reports should include only changes in this information):

- Description of sampling equipment
- Description of sampling procedures, including sampling times, frequencies, rates, and volumes
- Description of analytical procedures
- Description of calculational methods, for example, calculation of radionuclide quantities using gross radioactivity measurements
- Discussion of random and systematic error estimates, including methods of calculation and sources of systematic error
- Description of the calculation of the lower limit of detection
- Discussion of the program for ensuring the quality of results
- Description of calibration procedures
- Discussion of any unusual releases, including the circumstances of the release and any data available on the quantities of radionuclides released

- The basis for any determination that a stack or liquid release point need not be continuously sampled

The phrase "not detected" or similar phrases should not be used. Each reported result should be the following:

- A value and its associated standard deviation
- An indication that the result was below the stated value of the LLD

Self-Check Questions 8-5

INSTRUCTIONS: Fill in the missing words in each statement. Answers are located in the answer key section of the Trainee Guide. Choose from the following words.

adsorption	flowmeter	impaction	peroxide	removes
charcoal	granules	radioiodine	probe	representative
concentrate	higher	impingers	radon	syringe
constant	high-volume	papers	rate	tape

1. Air-moving systems for gaseous effluent sampling should maintain a _____airflow during sampling conditions.

2. The most commonly used method for collecting gases and vapors is_____.

3. Adsorbers are solid _____with large surface areas that are packed into cartridges or tubes.

4. Two of the most common adsorbers are activated _____and silica gel.

5. Absorption is performed by liquids inside devices called bubblers or _____.

6. Three common absorber solutions are ethylene glycol, hydrogen _____, and sodium hydroxide.

7. To avoid errors in determinations of the volumes of air sampled, it is extremely important that careful attention be paid to the use and calibration of the _____.

8. Particle _____ is the collection of particles that deviate from the airflow stream. It occurs when the stream bends as the airflow bypasses a solid object.

9. In general, the type of equipment typically needed to sample gases consists of a sampling _____, a sample delivery line, and a container to receive the sample.

10. Minute quantities of materials can be extracted from the air for trace contaminant analysis with specially designed _____samplers.

11. It is important to know the actual clock time at the start and stop and the elapsed time when conducting air sampling with _____air filters.

12. Temperature inversions cause airborne materials to _____near the earth.

13. High winds may cause dispersal and thus lower concentrations, but they may also pick material up and cause _____airborne levels.

14. Precipitation generally _____contaminants from the air.

15. One common form of interference in environmental air samples is natural _____. Its decay products collect on various media and provide additional alpha and beta activity.

16. Activated charcoal, which is used in charcoal canisters, is primarily used to collect _____.

17. Liquid reagents, chemically treated _____, and glass indicating tubes containing solid chemicals are three types of direct-reading colorimetric indicators that have been used for the determination of contaminant concentrations in air.

18. Detectors for real-time analysis may operate in a semicontinuous manner by filtering airborne particles onto a portion of continuous _____for a specified time period.

19. Airborne effluents should be continually sampled unless the license has established that radioactivity in the effluent is insignificant and periodic sampling is sufficient. If periodic sampling is used, the samples should be _____of actual releases.

20. The sampling _____at each release point should be such that a representative sample of the effluent is collected. The volumes of gaseous effluents should be reported so NRC staff can calculate quantities of radionuclide discharge.

Complete the following questions. Answers are located in the answer key section of the Trainee Guide.

21. What is a point source?

22. What is a diffuse source?

23. State three purposes for stack sampling.

24. What considerations should be addressed for stack sampling location?

25. What American National Standards Institute (ANSI) standard illustrates recommendations for multipoint sampling?

26. What is a normal sequence for a sampling train?

27. What safety precaution should be considered when an individual inserts a probe into a stack?

28. What are three major categories of filters and how are filters chosen for stack sampling?

29. In what areas in sampling lines are leaks likely to occur?

30. What are the requirements for gaseous sample analysis at nuclear fuel processing and fabrication processes?

31. Under what conditions should radionuclide analyses be more frequent?

32. The lower limit of detection for any analysis should not be more than what percentage of the concentration limits listed in Appendix B of 10 CFR Part 20?

33. For each release point, what information should be reported as appropriate?

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

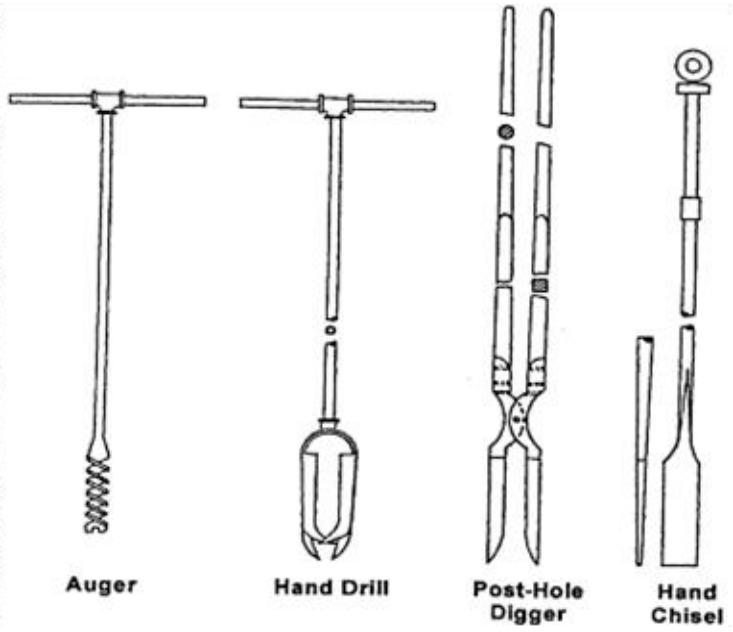
8.1.8 Identify techniques used in sampling solids.

SAMPLING SOLIDS

Compact Solids

Sampling of solids in compact form (in contrast to particulate form), such as soil or concrete, is accomplished by using digging and chiseling tools (i.e., augers, hand drills, post-hole diggers, or hand chisels). An auger sampler is a small helical screw or worm with a T-style handle. It is turned into the material and then pulled straight out. The material is knocked or scraped off with a spatula or other suitable tool into a receptacle and subsampled to form a sample. An auger and other hand tools with a similar application are illustrated in Figure 8-22, Equipment for Obtaining Samples of Compact Solids. This type of sampling is suitable where disturbed samples can be used and in taking advance samples before tube sampling during core drilling.

Figure 8-22. Equipment for Obtaining Samples of Compact Solids



An undisturbed sample can be withdrawn by using special techniques and equipment associated with drilling. Drilling equipment is used to provide a clean hole to permit the driving of a split-barrel sampler into the hole to remove the sample.

Material not suited to other methods of sampling may be broken up by using any kind of a sharp-edged tool, such as an axe, adz, pick, or chisel.

Particulate Solids

Particulate solids may be found in various conditions at the time of sampling. Appropriate tools for sampling in specific situations are discussed later in this section, as well as the sampling of airborne particulates.

Sampling from Hoppers, Drums, Bags, and Others

Tubular thief samplers can be inserted into material and held so that the free-flowing material falls out into a sample receptacle. Concentric tube thief samplers are designed so that holes are closed during insertion. After insertion to the proper depth, the insertion holes are opened by rotating the inner tube. After the inner tube is filled with the sample material, the inner tube is again rotated to close the holes and the sampler removed, with the sample still inside the inner tube.

Sampling from Conveyors or in Chutes or Free-Flowing Streams

Hand scoops can be used to draw samples by hand from a stationary conveyor belt. Samples collected from several points across the belt can be composited to obtain a more representative sample. The scoop may also be used to sample from the surface of drums, bags, barrels, or other containers. For sampling a moving conveyor, an automatic or semiautomatic device can be used. A mechanically operated head moves across the material flow at a preset interval and transfers the sample into a collecting system.

On-line sampling of process streams may be accomplished by equipment similar to that used for continuous analysis of liquids or gases. An on-line analyzer is interfaced with a digital computer. By using this method, materials can be analyzed more than 100 times faster than by hand sampling or conventional types of mechanical sampler.

CARE OF EQUIPMENT FOR SAMPLING SOLIDS

It is important that all sampling equipment be clean, dry, and free of contaminants and made of materials inert to the material being sampled. Care should be taken to remove all traces of cleaning agents from sample equipment. All empty containers should be kept covered. Sample containers should be labeled immediately after the sample is obtained; all pertinent information should be included in indelible marking and the label secured in a permanent fashion. Storage facilities should be adequate to preserve the samples from deterioration; refrigerated storage should be provided if required.

SOIL SAMPLING AND MEASUREMENT

Since soil represents an integrated sample of deposition, it can serve as an indicator of the movement of effluents and contaminants through the atmosphere or water.

Meteorology, Geography, and Demography

Soil contamination results from airborne deposition or liquid runoff (storm) on the land. Wind and rain can cause contamination on the land and into groundwater and surface water resources.

Population exposure pathway analysis determines the potential effect to man. Contamination can result in exposure to humans through a variety of ways; therefore, analysis of exposure pathway based on local demography is important.

Purpose of Project

The purpose of the project should dictate the type of sampling used. Factors to consider include the following:

- Total inventory
- Surface or subsurface sampling, potential for contamination
- Depth profile
- Uniformity of the contamination
- Accuracy required to provide useful results
- Minimization of cross-contamination

Site Characteristics

Site characteristics (such as soil type, topography, source, and current distribution of the contamination) impact soil sampling and measurement. For example, clay soils are very impervious to the movement of both water and radionuclides.

Sampling Sites

In order to accurately characterize soil contamination, sampling and measurement have to occur in the appropriate location and at the appropriate depth. The determination of soil sampling sites is an important aspect of the sampling plan. It is also important that the area has not been disturbed and that it is representative of the area/location being evaluated. A large area of collection is more preferable and assists in making the sample more representative. Other areas of importance are listed below:

- The expected distribution of contamination in the soil
- How the contamination entered the soil (i.e., either airborne or as a result of a spill)

Determination of Analytical Method

The determination of analytical method will depend on the chemical nature of the contaminant or effluent in question. The most useful measures of deposited material in soil relate to the concentration per mass. Sampling is carried out so that the weight of the materials collected provides a comparison with the area and depth sampled.

SOIL SAMPLING AND MEASUREMENT TECHNIQUES

Soil sampling and measurement techniques include:

- Surface sampling
- Subsurface sampling
- Sediment sampling
- In-situ exposure measurements

Surface Sampling

Sampling of surface soils generally includes soils down to 15 cm (6 inches).

Evaluation of suspected distribution methods is necessary for determination of an appropriate sampling method. For example, there are surface sampling techniques used to sample soils that have been contaminated by aerial deposition. This type of contamination is usually evenly distributed horizontally but stratified vertically. If the contamination has been deposited so distribution in the soil is heterogeneous, the use of a scoop or trowel is an acceptable method of collection.

Subsurface Sampling

Subsurface sampling methods are generally more labor intensive, require more specialized equipment, and are less precise than surface sampling. If the potential contamination is not of concern below 30 cm, bucket augers provide a convenient method of subsurface sampling.

Sediment Sampling

Sediment sampling provides an indication of the accumulation of certain undissolved radionuclides or other contaminants in the aquatic environment. Sediment samples can be collected manually (by hand in shallow water or by diving in deeper water) or mechanically (by dredge or with a core sampler).

In-Situ Gamma Measurements

Soil concentrations determined from in-situ gamma measurements (gamma ray spectrometry) are important indicators of direct radiation in the vicinity of a nuclear facility. Gamma measurements in the field can provide useful information, including the identification of radionuclides and determination of environmental exposure rates.

Instrumentation is available for in-situ gamma spectroscopy. The method is to put the detector over the sample, rather than the sample on the detector. This can be used to identify and quantify contamination without sampling. Calibration must compensate for surface penetration.

In-situ gamma measurements also offer an opportunity for determining soil concentrations at surface and subsurface ground locations. This provides rapid on-site analysis and reduces the cost associated with surveys. This is also an activity noted in various remedial action programs, such as the cleanup of uranium mill tailings covered in 40 CFR 192. Field measurements can define the spatial extent of the contamination and provide faster results, which impacts the cost of personnel and equipment.

In recent years, there have been significant developments in the area of in-situ gamma spectroscopic analysis. Energy attenuation can be evaluated based on apparent differences in abundances of known multiline emissions. Recent developments have made this a powerful real-time analytical method.

SOIL SAMPLING DEVICES

Soil sampling devices include the following tools:

- Scoops and shovels
- Augers (continuous flight and bucket augers)
- Tube samplers

Scoops and shovels are used to sample shallow, dry, or powdered material or soil. They are like the ones used for gardening.

An auger consists of sharpened spiral blades attached to a hard metal central shaft. An auger samples hard or packed solid wastes or soil.

"Thin walled metal tubes can be used to recover relatively undisturbed soil samples suitable for laboratory analysis . . . thin walled tubes may be used in piston, plug, or rotary-type samplers . . .," American Society for Testing and Materials (ASTM) Designation: D 1587-83, Standard Practice for Thin-Walled Tube Sampling of Soils.

PACKAGING SOIL SAMPLES

Samples can be bagged (or double-bagged) in plastic, taped, and then transferred to metal cans. Where organic contaminants are to be analyzed, precleaned glass containers will be used. These should be fitted with Teflon or aluminum foil liners.

If a carrier is to be used to transport a large number of soil samples, it should be packed into a strong container, such as a 5- to 10-gallon steel drum. If the container cannot be locked, it should be fitted with a security seal.

LABELING REQUIREMENTS

Soil sample labeling requirements include a unique sample identifier and the date and time, as well as information establishing the location and depth of the sample. Additionally, a soil sampling data sheet may be used to identify soil characteristics that could impact sampling and measurement.

All soil samples should be kept within eyesight or stored in a locked box or vehicle at all times.

SOIL SAMPLING PREPARATION

Methods for preparing soil samples vary from facility to facility. Samples are placed on dry ice in a cooler for preservation. Soil preparation is mainly accomplished through drying and homogenizing the sample for analysis.

The preparation process will be specific to the purpose of the analysis. Typically the following process is used:

- The sample is dried (air- or oven-dried).
- The total weight is measured.
- Large rocks may be removed by hand.
- The sample is crushed and blended (mortar and pestle, ball mill, or bagging and crushing with a hammer).
- The sample may be sieved prior to analysis, depending on data end use.

At times a stratified sample is required rather than a homogenized sample. Core sampling is a process for recovering stratified soil samples for analysis.

ANALYTICAL TECHNIQUES

The types of analysis conducted on soil samples include the following techniques:

- Gamma and alpha spectroscopy/spectrometry
- Solvent extraction process for nonradiological contaminants

Soil and sediment are typically dried and homogenized (mixed) prior to any analyses. Any alpha or beta analyses on soil require further processing and purification. Dry material is ground and/or sieved to increase homogeneity. Ashing may be required to destroy organic material. This is followed by chemical analysis for specific alpha or beta emitters. Results are activity per unit of dry weight.

It is also necessary to sample and measure other **nonradiological contaminants**. Organic solvents are often used to extract the pollutants from the sample medium. The analysis is then performed on the concentrated extract.

Data Interpretation

Soil concentrations are usually reported in units of activity per unit mass. Care must be taken during interpretation to ensure that sampling and measurement are representative of the population. For example, a rocky soil may have the same reported concentration as a sandy soil but a much lower total activity per unit volume or area.

Soil moisture and rocks add to the mass of the soil but do not contribute activity. To compensate for this, the volume and weight of rocks are determined first and the materials removed prior to the preparation process for analysis. Soil moisture content varies depending on the weather; therefore, the soil is usually dried prior to analysis. The method used to dry the soil will affect the reported concentration. For example, oven-dried soils may weigh 10% less than air-dried soils.

Factors Important in Soil Sampling

Factors that are important in soil sampling and measurement include the following:

- The sample is representative of the media being analyzed.
- Soil sampling is the primary means for evaluating the environmental inventory of radioisotopes.
- Soil represents an integrated sample of airborne deposition.
- Soil serves as a more sensitive indicator of movement through the atmosphere than air sampling.

Often not enough emphasis is placed on the importance of proper sampling methods to accurately represent the total pollutant being sampled. Because of this, it is very important that a well-designed plan be developed that takes into consideration the uniformity of the contamination, the required accuracy necessary to provide reasonable results, and the minimization of cross-contamination. Just how representative the sample(s) is of the total contamination is a very important issue in soil sampling.

SAMPLING AND MEASUREMENT OF BIOTA

Critical pathways of exposure for population groups living within the vicinity of fuel cycle facilities include the biota. Aquatic plants and animals can be vectors in this water-plant-animal-human pathway. Aquatic organisms should be sampled and analyzed at least annually to provide compliance with the established limit.

Freshwater food sources (fish, shellfish, and water fowl) should undergo preliminary pathway analysis to determine whether they represent a source of contamination in the population exposure pathway of known effluents/contaminants. The migratory nature of some of these species may complicate the analysis.

Sampling Techniques and Devices

Techniques and devices used for sampling of biota depend on what is being sampled. Following is a list of aquatic plants and animals and devices that may be used to obtain samples.

- Fish may be collected by nets, rods and reels, or electrocution. They may also be purchased through commercial fishing sources.
- Shellfish can be gathered or purchased commercially from sources in the vicinity of the site.
- Waterfowl, such as ducks and geese, are generally trapped or
- hunted for collection.
- Marine foods present a problem in the determination of the exact source of contamination. It is usually possible to purchase seafood that has been harvested from local sources.

Generally speaking, collection of the various species is accomplished through hunting, fishing, trapping, or purchasing locally harvested samples from commercial businesses. The gathering of aquatic plants is accomplished through pulling up or cutting plants for collection and placing the materials in large plastic bags.

When Sampling Is Indicated

When sampling is indicated, the following apply:

- Species are collected in sufficient quantity and from appropriate locations to be considered representative.
- Background characterization from samples unaffected by effluents/contaminants are collected.
- The sampling location for contaminants/effluents should be located downstream of the discharge point(s), where the water is well mixed.
- Wildlife agencies should always be contacted.

Handling and Packaging

Fish and shellfish are placed in plastic bags, sealed, and properly labeled, then placed under dry ice before delivery to the laboratory for analysis. The handling and packaging of biota depend on the species being sampled and the method of collection. Generally speaking, it is appropriate to preserve samples by refrigeration or the addition of an appropriate preservative until they can be freeze-dried. Samples are weighed, freeze-dried, and pulverized prior to analysis for radioactive and other contamination. The results are an indication of the concentration of the contaminant within a given volume of material. The parts sampled should be parts that would be eaten by humans.

Aquatic plants must be gathered in large quantities, because most of their weight is water and drying greatly diminishes their weight. The roots are either rinsed clean or removed by trimming in the field. They then are placed in plastic bags.

SAMPLING AND MEASUREMENT OF VEGETATION

Samples of pathway-significant agricultural products grown around sites/facilities should be collected for analysis to determine the presence of effluents or other contaminants from waste stream discharges. If the preliminary analysis indicates that the annual effective dose/exposure equivalent from ingestion of these foods is above acceptable limits, then analysis should be completed to determine the ingestion dose/exposure.

One agricultural product commonly monitored is vegetation. Vegetation includes vegetables, grains, and fruit. Potential doses to humans determined through pathway analysis will indicate the species to sample.

Purpose of Collection

Collection and analysis of vegetation serve three purposes:

- Evaluation of potential radiation doses received by humans
- Prediction of the possible concentrations in meat, eggs, and milk from animals consuming contaminated forage
- Monitoring trends in environmental contamination and possible long-term accumulation of radionuclides and other contaminants

Collection Issues

Samples collected will depend on the species available, seasonal growth patterns, farming practices, and the reasons for sample collection.

When measurement of radioactivity is below minimum detectable concentrations, dose calculations should include an estimate of potential contribution.

Collection Sources

Sample collection locations should be within the potential pathways for biota effects. Collection occurs from a variety of sources, including commercial, local farms, and family gardens. Vegetation should come from open areas where deposition would be normal.

Techniques and Devices

Samples are gathered by cutting the foliage or grass and placing the sample materials in plastic bags. The materials are cut rather than pulled up to keep dirt and the roots from getting into the sample. The parts of the plants that need to be sampled and measured are those that are part of the exposure pathway. For example, the part of grass that the cow eats is the part that should be collected.

Handling and Packaging

Vegetation for sampling is gathered in quantities that have sufficient weight after drying to conduct the analyses. The samples are usually freeze-dried and pulverized prior to analysis. It is important to gather the edible parts of the plant. Grass is sampled by cutting it off at ground level with scissors.

SAMPLING AND MEASUREMENT OF THE FOOD CHAIN

Samples of agricultural products grown around sites/facilities should be collected and analyzed for contamination and presence of radionuclides from site operations. Fresh produce, meat, poultry, and eggs can be purchased from local producers and analyzed for effluents/contaminants. Milk is widely consumed by all age groups and is frequently one of the most important foods contributing to the radiation dose to people from animals pastured near a nuclear site.

Samples should also be taken from a background location unlikely to be affected by the site/facility. This location should be within close proximity to the site.

Sampling Techniques and Devices

Techniques and devices used for sampling and measurement will be determined by the material being sampled.

Sampling of Milk

Considerations for sampling of milk:

- The number of locations depends on the distribution of cows in the vicinity.
- Annually samples should be taken from one background and one potentially affected location within the vicinity of the site.
- Information about dates and distribution patterns of local milk production is important for analytical results to be meaningful.

- Samples of raw milk should be collected and analyzed.
- Guard against contamination and spoiling of the milk.
- Techniques should be within the protocol of accepted appropriate state agricultural standards.

Sampling of Meat

Considerations for sampling of meat:

- Because of a time delay in the transfer of radionuclides from the point of release to meat, meat samples are not useful for time correlation of releases.
- Sampling is required only to evaluate the radiation doses received via foodstuff.
- If C14 is the predominant radionuclide in the environment, the dose from inhalation and external exposure would be small compared to the dose from ingestion of foods.
- Preliminary pathway analysis will determine whether it is necessary to frequently sample meat.
- When sampling is indicated, it is better to sample at slaughterhouses or farms to avoid further delays.
- Feed consumption rates will impact the uptake of effluents and contaminants in animals; for example, chickens with a low feed consumption rate, would be less likely to have high levels than beef cattle (with a higher feed consumption rate).

Sampling of Eggs

Considerations for sampling of eggs:

- Eggs from chickens that are allowed to roam are more likely to have detectable amounts of effluent radionuclides.
- Preliminary pathway analysis will determine whether frequent sampling is indicated.
- The sample size should be about a dozen large eggs.

Sampling of Game Animals

Considerations for sampling of game animals:

- Squirrel, deer, rabbits, and game birds are often part of the diets of certain individuals.
- Preliminary pathway analysis will determine whether sampling is necessary.
- Wild game can be trapped, acquired by hunters, collected after accidental road kills, or obtained from an appropriate state agency.
- Care should be taken not to cross-contaminate from internal and external sources on the sample.

Handling and Packaging

Meat should be placed in plastic bags, sealed, and properly labeled before delivery to the laboratory for analysis. Samples are generally freeze dried, pulverized, and analyzed according to procedures required for the effluent/contaminant being sampled. Collection of the parts for analysis that would be consumed is a requirement for appropriate sampling.

Generic Environmental Sampling and Measurement Plan

Example 8-4, "Examples of a Generic Environmental Sampling and Measurement Chart with Locations," provides you with a look at information, in tabular form, that needs to be addressed in the sampling plan and facility licenses or certifications. The information should be organized logically and provide an indication of the types of activities and locations associated with sampling and measurement at the facility.

In summary, factors that impact environmental sampling and measurement include the following:

- Proximity to the site/facility
- Types of contaminants/effluents being released in waste stream discharges from the plant
- Samples that are as representative of the location/area as possible
- Population exposure pathway

Example 8-4. Generic Environmental Sampling and Measurement Chart with Locations

EXAMPLE OF A GENERIC ENVIRONMENTAL SAMPLING AND MEASUREMENT CHART WITH LOCATIONS

Sample Type	Approximate Number of Samples and Locations	Collection Frequency	Analysis Types and Frequency
Fish and shellfish	1 each of the principal edible types from vicinity of outfall	Semiannually	Gamma isotopic analysis semiannually on edible portions
	1 each of the sample types from area not influenced by the discharges		
Fruits and vegetables	1 each of the principal food products grown near the point of maximum predicted annual ground concentration from stack releases and from any area that is irrigated by water in which liquid plant wastes have been discharged		
	1 each of the same foods grown at greater than 20 miles' distance in the least prevalent wind direction		
Meat and poultry	Meat, poultry, and eggs from animals fed on crops grown within 10 miles of the facility at the prevailing downwind direction or where drinking water is supplied from a downstream source		
Quality control	Samples as required for accurate sampling and analysis		

EXAMPLE OF A GENERIC ENVIRONMENTAL SAMPLING AND MEASUREMENT CHART WITH LOCATIONS

Sample Type	Approximate Number of Samples and Locations	Collection Frequency	Analysis Types and Frequency
Drinking water	Any supplies obtained within 10 miles of the facility that could be affected by its discharges or the first supply within 100 miles if none exists within 10 miles		
Sediment, benthic animals, and aquatic plants	1 directly downstream of outfall	Semiannually	Gamma isotopic analysis semiannually
	1 upstream of outfall		
	1 at dam site downstream or in impoundments		
Soil	Soil samples are generally taken from the same site locations that air samples are recovered from.		
	Off-site soil sample locations should reflect the area surrounding the facility.		
Food chain			
Milk	1 sample at nearest off-site dairy farm in the prevailing downwind direction	Monthly	Gamma isotopic analysis and radiostrontium analysis monthly

EXAMPLE OF A GENERIC ENVIRONMENTAL SAMPLING AND MEASUREMENT CHART WITH LOCATIONS

Sample Type	Approximate Number of Samples and Locations	Collection Frequency	Analysis Types and Frequency
Air particulates	1 sample from several locations of the highest off-site ground-level concentrations	Continuous collection - filter change as required	Isotopic analysis and composite quarterly
	1 sample from the nearest residence		
	1 sample from the nearest community		
	1 sample from a location greater than a 20-mile radius in the least prevalent annual wind direction		
	1 sample from 3-5 locations on the fenceline (several of these should be placed in the predominant wind direction)		
Surface water	1 upstream	Monthly	Gross beta, gamma isotopic analysis monthly, composite for --- quarterly
	1 downstream after dilution (e.g., 1 mile)	(Record status of discharge operations at time of sampling)	
Ground water	1 or 2 from sources most likely to be affected		

Self-Check Questions 8-6

INSTRUCTIONS: Complete the following questions. Answers are located in the answer key section of the Trainee Guide.

1. What equipment can be used for sampling solids in compact form, such as soil or concrete?

2. When sampling from hoppers, drums, and bags, tubular thief samplers can be used. How do they work?

3. Why might it be a good practice to collect samples from several points across a stationary conveyor belt?

4. Since soil represents an integrated sample of deposition, it can serve as what kind of indicator?

5. In order to accurately characterize soil contamination, what sampling and measurement considerations should be addressed?

6. How are subsurface sampling methods different from surface sampling?

7. What can sediment sampling indicate?

8. What are some advantages of in-situ gamma measurements?

9. How can soil samples be packaged?

10. How often should aquatic organisms be sampled and analyzed?

11. What do the handling and packaging of biota depend upon?

12. What are the three purposes of vegetation collection and analysis?

13. What are some considerations for collecting and analyzing vegetation?

14. What is the most widely consumed food contributing to the radiation dose to people from animals pastured near a nuclear site?

15. What food product samples should be taken to determine the presence of radionuclides in the food chain?

16. What factors impact environmental sampling and measurement?

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

8.1.9 Identify common sampling and measurement concerns related to sample collection and handling.

8.1.10 Identify measurement program concerns.

8.1.11 Identify sampling and measurement concerns for maintaining nuclear criticality safety.

SAMPLE COLLECTION

Nonrepresentative sampling can occur during sample collection due to improper selection of sample location, inconsistent sampling techniques, and sampling equipment failure. Table 8-4 lists selected sampling and measurement problems and prevention measures related to sample collection.

Table 8-4. Selected Sample Collection Problems and Prevention Measures

Problem	Prevention
Sampling location not representative	◙ Determine parameter(s) to be analyzed prior to sampling ◙ Design sampling program based on detailed understanding of pathway analysis ◙ Carefully select background locations ◙ Avoid locations where interference exists ◙ Determine accessibility to sampling point ◙ Check flow direction
Sampling location cannot be reoccupied	◙ Maintain detailed logs of field activities ◙ Implement procedures to permanently mark sample locations

Table 8-4. Selected Sample Collection Problems and Prevention Measures

Problem	Prevention
Sample not representative of medium at selected location	◙ Design sampling program based on understanding of effluent or environmental processes ◙ Conceptualize and assess homogeneity of target medium - consider need for (flow-weighted) composite sample, continuous sample, or stratification of medium ◙ Ensure that selected equipment provides type of sample required ◙ Sample in strict accordance with SOPs
Measurement results show excessively high variability	◙ Ensure replicate samples are collected from a homogeneous area ◙ Sample in strict accordance with SOPs ◙ Consistently implement equipment decontamination procedures
Measurement results are not accurate (i.e., biased)	◙ Eliminate all field sources of systematic contamination ◙ Sample in strict accordance with SOPs ◙ Implement rigorous field equipment calibration program
Measurement results are not comparable over space and/or time	◙ Ensure that the same sampling equipment and SOPs are employed throughout the program
Improper connections and defective valves or pigtails	◙ Conduct leak rate testing and cold pressure testing prior to sampling
Release of UF_6 at an autoclave	◙ Ensure the availability of sampling interlocks to prohibit the opening of both the block inlet and sample outlet valves while the autoclave heaters are on
Cross-contamination between subsequent samples	◙ Flush equipment with an aliquot of the material to be sampled

Table 8-4. Selected Sample Collection Problems and Prevention Measures

Problem	Prevention
Nonhomogeneity	◙ Ensure homogeneity of liquids and solids prior to sampling
Insufficient purging of sampling lines	◙ Conduct test procedures for adequate purging times
Errors due to anisokinetic sampling ◙ Failure to withdraw a sample from a flowing stream at the same velocity as that which exists locally in the stream will result in non-representative sampling ◙ If the sampling rate is much higher than the local stream velocity, a greater fraction of smaller rather than larger particles will be drawn into the probe ◙ If sampling rate is much lower than the stream velocity, large particles will be impacted into the collecting probe	◙ Withdraw sample at appropriate velocity ◙ If the flow rate changes by more than + 10 percent during collection of a sample, a correction should be made by averaging the initial and the final flow rates.

SAMPLE HANDLING

Good sample handling practices are important for prompt analysis and review of sample data that could indicate degrading operating conditions or the loss of barriers that protect workers, the public, and the environment. Essential to timely analysis is a formalized process embodied in procedures that provide clear definition of responsibilities and ownership for sample data review and analysis and appropriate requirements for timeliness. Specific criteria providing early-warning points during data analysis can also enhance prompt detection of abnormal conditions. Table 8-5 lists selected sampling and measurement problems and prevention measures related to sample handling.

Table 8-5. Selected Sample Handling Problems and Prevention

Problem	Prevention
Quality of samples has been compromised during handling	◙ Ensure custody in the field ◙ Prepare samples for shipment according to SOPs ◙ Include appropriate quality control samples to identify sources of contamination

Table 8-5. Selected Sample Handling Problems and Prevention

Problem	Prevention
Contamination of the sample after removal from the bulk material	◙ Adhere to good sample handling practices and procedures
Failure of the operator to follow procedures	◙ Check procedures for accuracy and completeness ◙ Conduct training
Chemical and physical changes in the material during sampling Example: Plutonium dioxide powder can gain 1% in weight within a few hours if exposed to an increase in humidity.	◙ Establish and maintain careful control over conditions when sampling material, particularly if it is to be relocated prior to sample analysis
Laboratory cannot identify one or more samples	◙ Develop and implement a data management plan and sample identification scheme ◙ Implement rigorous sample labeling and chain-of-custody procedures
Two samples arrive at the laboratory with the same ID	◙ Develop and implement a data management plan and sample identification scheme ◙ Implement rigorous sample labeling procedures
Measurement results show excessively high variability	◙ Ensure field subsamples are drawn from a thoroughly mixed sample
Measurement results show bias	◙ Eliminate systematic sources of error in sample handling through rigorous implementation of SOPs
Errors in labeling, data entry, errors in transfer of data from lab books to computer files	◙ Double-check labeling of containers ◙ Double-check accuracy of data recording and transfer data

MEASUREMENT PROGRAM

An adequate measurement program should be implemented and maintained for measuring the quantities of special nuclear material or source material that is received, produced, transferred,

inventoried, shipped, discarded, or otherwise removed from inventory. The licensee measurement program should be thoroughly tested and evaluated before its use and should be continually controlled to a level of effectiveness sufficient to satisfy the capabilities required for detection, response, and accounting. Table 8-6 lists selected sampling and measurement problems and prevention measures related to measurement programs.

Table 8-6. Selected Measurement Program Problems and Prevention

Problem	Prevention
Inadequate weight measurement controls	◙ Reverify primary and secondary weight standards annually ◙ Conduct routine maintenance and calibration every six months ◙ Verify check weights quarterly ◙ Ensure all accountability scales are checked each operating shift against weight standards traceable to the U.S. Bureau of Standards
Inadequate analytical measurement controls	◙ Use reference standards traceable to the national system ◙ Use working standards traceable to reference standards ◙ If control limits are exceeded, recalibrate standards ◙ Conduct daily analyses of check samples
Measurement results show excessively high variability	◙ Measure in strict accordance with SOPs ◙ Consistently implement equipment decontamination procedures ◙ Ensure laboratory subsamples are drawn from a thoroughly mixed sample ◙ Anticipate presence of interfering substances
Measurement results are not accurate (i.e., biased)	◙ Eliminate all sources of systematic laboratory contamination ◙ Measure in strict accordance with SOPs ◙ Implement rigorous laboratory equipment calibration program ◙ Include appropriate quality control samples (e.g., spikes)
Measurement results are not representative of parameter of interest	◙ Plan sample processing activities (e.g., filtering or not filtering) so requisite data will be produced ◙ Strictly apply processing SOPs

Table 8-6. Selected Measurement Program Problems and Prevention

Problem	Prevention
Detection limits are above levels of concern	◙ Plan activities so measurement technique has requisite sensitivity ◙ Ensure adequate sample volume is available
Sample in process cannot be identified or is misidentified	◙ Develop and implement a data management plan and sample identification scheme ◙ Implement rigorous sample labeling and chain-of-custody procedures
Quality of laboratory measurements not established, potentially resulting in misapplication of data	◙ Conduct rigorous data validation program

SAMPLING AND MEASUREMENT FOR MAINTAINING NUCLEAR CRITICALITY SAFETY

Sampling and measurement to maintain nuclear criticality safety require that fissionable materials are produced, processed, stored, transferred, disposed of, or handled in such a manner that the probability of a criticality incident is acceptably low. Compliance with proper handling and storage requirements of special nuclear materials is an essential part of criticality control measures, and personnel must be aware of the importance of sample and measurement verification when performing operations involving fissile material. Table 8-7 lists selected sampling and measurement problems and prevention measures related to nuclear criticality safety.

Table 8-7. Selected Nuclear Criticality Safety Problems and Prevention

Problem	Prevention
Failure of the operator to follow procedures	◙ Check procedures for accuracy and completeness ◙ Conduct training
Failure of controlling enrichment or concentration of fissile isotopes for criticality safety	◙ Determine subcriticality before new operations are started or existing ones are changed ◙ Sample prior to transfer of solutions
Failure of neutron poisons as criticality control	◙ Verify concentrations of neutron poisons by sampling
Undissolved solids, air sparging, and stirring effects	◙ Verify geometric control limits ◙ Ensure proper procedures ◙ Design considerations: □ A single gas sparger may be used for vessels up to 3 meters in diameter; larger vessels should have more than one sparger □ Use of Raschig rings in vessels should be avoided wherever possible. Other means of preventing criticality should be considered for such vessels. If Raschig rings are used, precautions should be taken to ensure thorough mixing and completeness of transfer. Because Raschig rings tend to compact during use, consider the need for more frequent calibration of vessels filled with Raschig rings. □ If mixing is accomplished by external recirculation and if the recirculating pump and piping are not dedicated to the measurement vessel, care should be exercised that the solution to be measured is completely returned to the measuring vessel and that none escapes elsewhere in the process.

Self-Check Questions 8-7

INSTRUCTIONS: Complete the following questions. Answers are located in the answer key section of the Trainee Guide.

1. Why can nonrepresentative sampling occur during sample collection?

2. Why are good sample handling practices important?

Match the following sample collection problems in column A with prevention measures listed in column B.

Column A **Problem**	**Column B** **Prevention Measure**
A. Sampling location not representative	3.____Ensure replicate samples are collected from a homogeneous area
B. Measurement results show excessively high variability	4.____Conduct test procedures for adequate purging times
C. Improper connections and defective valves or pigtails	5.____Determine parameter(s) to be analyzed prior to sampling
D. Insufficient purging of sampling lines	6.____Conduct leak rate testing and cold pressure testing prior to sampling

7. The following three sampling and measurement problems could occur when handling samples. Describe possible prevention activities that could help to overcome each problem.

   A. Quality of sample has been compromised during handling

      Prevention:

   B. Failure of the operator to follow procedures

      Prevention:

   C. Measurement results show excessively high variability

      Prevention:

Match the following measurement problems in column A with prevention measures listed in column B.

Column A **Problem**	**Column B** **Prevention Measure**
A. Inadequate weight measurement controls	8.____Plan activities so measurement technique has requisite sensitivity
B. Detection limits are above levels of concern	9.____Develop and implement a data management plan and sample identification schema
C. Sample in process cannot be identified or is misidentified	10.___Reverify primary and secondary weight standards annually

11. What are four sampling and measurement problems that could occur when trying to maintain nuclear criticality safety?

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

8.1.12 List applicable regulations and requirements for sampling and measurement of in-process operations, effluents and discharges and within the environs of fuel cycle facilities.

APPLICABLE REGULATIONS AND REQUIREMENTS

Table 8-8, Regulatory Basis, is a list and description of regulatory documents that provide the foundation for national policies and goals relating to the protection and care of workers, the public, and the environment. This list does not include all pertinent regulatory documentation, only some common ones.

Table 8-8. Regulatory Basis

Document	Description
National Environmental Policy Act (NEPA) P.L. 91-190	◙ Basic policy-setting federal law relating to protection of the environment. Requires federal government agencies, such as the NRC, to prepare a detailed statement on the environmental effects of proposed federal actions significantly affecting the quality of the human environment. ◙ The NRC implements NEPA in Title 10 Code of Federal Regulations (CFR) Part 51 and Title 40 CFR Parts 1500-1508.
Title 10 CFR Part 20, "Standards for Protection Against Radiation"	◙ Establishes standards for protection against radiation hazards arising from activities under licenses issued by the NRC. Regulations apply to all persons who receive, possess, use, or transfer licensed materials. ◙ States that licensees shall make every reasonable effort to maintain radiation exposure, and releases of radioactive materials in effluents to unrestricted areas, as far below the limits specified in Title 10 CFR Part 20 as is reasonably achievable.

Table 8-8. Regulatory Basis

Document	Description
Title 10 CFR Part 40, "Domestic Licensing of Source Material"	◙ Establishes the procedures and criteria for the issuance of licenses to receive title to, possess, use, transfer, or deliver source material. ◙ Provides that the NRC may incorporate in any source material license such terms and conditions as it deems appropriate or necessary to protect health.
Title 10 CFR Part 50, "Licensing of Production and Utilization Facilities"	◙ Requires that licensees establish an appropriate surveillance and monitoring program to provide data on quantities of radioactive material released in liquid and gaseous effluents and to provide data on measurable levels of radiation and radioactive materials in the environment.
Title 10 CFR Part 70, "Domestic Licensing of Special Nuclear Material"	◙ Establishes procedures and criteria for issuance of licenses to receive title to, own, acquire, deliver, receive, possess, and initially transfer special nuclear material. ◙ Establishes and provides for the terms and conditions upon which the NRC will issue licenses.
Title 10 CFR Part 71, "Packaging and Transportation of Radioactive Materials"	◙ Regulates shipping containers and the safe packaging and transportation of radioactive materials under authority of the NRC and the Department of Transportation (DOT).
Title 10 CFR Part 72, "Licensing Requirements for Independent Storage of Spent Nuclear Fuel and High-Level Radioactive Waste"	◙ Establishes requirements, procedures, and criteria for the issuance of licenses to receive, transfer, and possess power reactor spent fuel and other radioactive materials associated with spent fuel storage in an independent spent fuel storage installation. ◙ Establishes criteria for the issuance of licenses to DOE for waste storage in a monitored retrievable storage installation.
Title 10 CFR Part 74, "Material Control and Accounting of Special Nuclear Material"	◙ Establishes the requirements for the control and accounting of special nuclear material at fixed sites and for documenting the transfer of special nuclear materials. ◙ Establishes the control and accounting of source material at enrichment facilities.
Title 10 CFR Part 76, "Certification of Gaseous Diffusion Plants"	◙ Establishes procedural requirements, generally applicable NRC health and safety standards, technical safety requirements, and safeguards and security requirements specific to gaseous diffusion plants.

Table 8-8. Regulatory Basis

Document	Description
Title 30 CFR Part 828, "Special Permanent Program Performance Standards - In-Situ Processing"	◙ Ensures that all in-situ processing activities are conducted in a manner that preserves and enhances environmental values in accordance with the Act. ◙ Provides additional performance, reclamation, and design standards to reflect the nature of in-situ processing.
Title 40 CFR Part 190, "Environmental Standards for Nuclear Power Operations"	◙ Requires that maximum annual radiation dose to individual members of the public resulting from fuel cycle operations be limited to 25 millirems to the whole body and to all organs except the thyroid, which is limited to 75 millirems. This impacts uranium mill tailing operations.

Regulatory Guidance

The purpose of regulatory guides is to provide assistance to applicants in specific requirements for various types of activities associated with the nuclear industry. Table 8-9 lists regulatory guides that address sampling and measurement issues related to in-process operations, effluents and discharges and environmental surveillance. This list is not meant to be totally inclusive of all applicable regulatory guides, but provides a basis to explore additional relevant materials.

Table 8-9. Regulatory Guides

Regulatory Guide	Description
3.5 Standard Format and Content of License Application for Uranium Mills (for comment) (Draft WM 039-4, Proposed Revision 2, published August 1981)	◙ Provides specific guidance on the format and content of an application for an NRC source material license authorizing uranium milling activities ◙ References other NRC regulatory guides necessary to prepare specific sections of the application
3.8 Preparation of Environmental Reports for Uranium Mills	◙ Identifies information needed by the NRC staff to assess the potential environmental effects of the proposed uranium mill and directly associated mining activities ◙ Establishes a format acceptable to the NRC staff for its presentation ◙ Helps to ensure the completeness of the information provided ◙ Assists the NRC staff and others in locating the information ◙ Aids in shortening the time needed for the review process

Table 8-9. Regulatory Guides

Regulatory Guide	Description
3.46 Standard Format and Content of License Applications, Including Environmental Reports, for In Situ Uranium Solution Mining (Draft FP 818-4, published July 1980)	◙ Provides instructive guidance to a licensee applying to operate an in-situ uranium solution mining operation ◙ Provides guidance on the development of the environmental report that discusses the operation's impact on the health and safety of the public and on the environment
3.52 Standard Format and Content for the Health and Safety Sections of License Renewal Applications for Uranium Processing and Fuel Fabrication	◙ Provides specific guidance for the preparation of the health and safety sections of license renewal applications ◙ Recommended by the NRC for use in uranium processing and fuel fabrication renewal applications
3.55 Standard Format and Content for the Health and Safety Sections of License Renewal Applications for Uranium Hexafluoride Production	◙ Identifies the type and quality of information needed in an application for license renewal ◙ Recognizes that the physical size, process scope (chemical or mechanical), and plant capacity all have a bearing on the complexity and level of license application detail
3.65 Standard Format and Content of Decommissioning Plans for Licensees Under 10 CFR Parts 30, 40, and 70 (DRAFT CE 304-4, published December 1985)	◙ Is applicable to licensees when they decide to permanently discontinue all licensed activities involving nuclear materials ◙ Identifies the information needed by the NRC staff for evaluations involving decommissioning ◙ Provides a format for submitting this information
4.9 Preparation of Environmental Reports for Commercial Uranium Enrichment Facilities	◙ Provides assistance to applicants for the development of environmental reports dealing with the construction, operation, and decommissioning of uranium enrichment facilities
4.14 Radiological Effluent and Environmental Monitoring at Uranium Mills	◙ Describes programs acceptable to the NRC staff for measuring and reporting releases of radioactive materials to the environment from typical uranium mills
4.15 Quality Assurance for Radiological Monitoring Programs (Normal Operation) - Effluent Streams and the Environment	◙ Describes a method acceptable to the NRC staff for designing a program to ensure the quality of the results of measurements of radioactive materials in effluents and the environment outside nuclear facilities during normal operations

Table 8-9. Regulatory Guides

Regulatory Guide	Description
4.16 Monitoring and Reporting Radioactivity Releases of Radioactive Materials in Liquid and Gaseous Effluents from Nuclear Fuel Processing and Fabrication Plants and Uranium Hexafluoride Production Plants	◙ Describes methods acceptable to the NRC for developing effluent monitoring programs in license applications for monitoring and reporting effluent data by licensees ◙ Is applicable to nuclear fuel processing and fabrication plants and uranium hexafluoride production plants
5.4 Standard Analytical Methods for the Measurement of Uranium Tetrafluoride (UF4) and Uranium Hexafluoride (UF6)	◙ Identifies acceptable methods for subsampling and chemical and isotopic analysis of uranium tetra- and hexafluoride which an applicant may specify as part of her/his procedures for accounting for special nuclear material
5.5 Standard Methods for Chemical, Mass Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets	◙ Identifies acceptable methods for chemical, isotopic, and impurity analysis which an applicant may specify as part of her/his procedures for accounting of special nuclear material
5.8 Design Considerations for Minimizing Residual Holdup of Special Nuclear Material in Drying and Fluidized Bed Operation	◙ Describes acceptable design features and characteristics for minimizing the residual holdup of special nuclear material in drying and fluidized bed operations after shutdown, draindown, or cleanout in order to facilitate material control and accountability procedures
5.9 Guidelines for Germanium Spectroscopy Systems for Measurement of Special Nuclear Material	◙ Is intended both to provide some general guidelines acceptable to the NRC staff for the selection of germanium spectroscopy systems and to point out useful resources for more detailed information on their assembly, optimization, and use in material protection measurements
5.11 Nondestructive Assay of Special Nuclear Material Contained in Scrap and Waste	◙ Details procedures acceptable to the NRC staff to provide a framework for the use of NDA in the measurement of scrap and waste components generated in conjunction with the processing of special nuclear material
5.21 Nondestructive Uranium-235 Enrichment Assay by Gamma Ray Spectrometry	◙ Describes conditions for U-235 enrichment measurements using gamma ray spectrometry that are acceptable to the NRC staff and provides procedures for operation, calibration, error analysis, and measurement control
5.25 Design Considerations for Minimizing Residual Holdup of Special Nuclear Material in Equipment for Wet Process	◙ Describes basic design features and characteristics acceptable to the NRC staff for minimizing the residual holdup of special nuclear material after draindown or

Table 8-9. Regulatory Guides

Regulatory Guide	Description
Operations	cleanout of equipment used in wet process operations
5.37 In Situ Assay of Enriched Uranium Residual Holdup	◙ Describes procedures acceptable to the NRC staff for the in situ assay of the residual enriched uranium holdup
5.38 Nondestructive Assay of High-Enrichment Uranium Fuel Plates by Gamma Ray Spectrometry	◙ Describes features of a gamma ray spectrometry system acceptable to the NRC staff for nondestructive assay of high-enrichment uranium fuel plates or fuel plate core compacts
5.39 General Methods for the Analysis of Uranyl Nitrate Solutions for Assay, Isotopic Distribution, and Impurity Determinations	◙ Identifies methods acceptable to the NRC staff for chemical, isotopic, and impurity analyses which an applicant may specify as part of her/his procedures for accounting for special nuclear material.
5.48 Design Considerations -- Systems for Measuring the Mass of Liquids	◙ Pertains to design considerations for methods of measuring the mass of liquid contained in a vessel and identifies those design considerations which the NRC staff considers to be adequate for minimizing the error associated with that measurement
5.53 Qualification, Calibration, and Error Estimation Methods for Nondestructive Assay	◙ Describes methods and procedures acceptable to the NRC staff for meeting the provisions of paragraph 70.58(f) of 10 CFR Part 70 as it relates to the use of nondestructive assay
5.58 Considerations for Establishing Traceability of Special Nuclear Material Accounting Measurements	◙ Presents conditions and procedural approaches acceptable to the NRC staff for establishing and maintaining traceability of special nuclear material control and accounting measurements
8.37 ALARA Levels for Effluents from Material Facilities	◙ Provides guidance on designing an acceptable program for establishing and maintaining as low as reasonably achievable (ALARA) levels for gaseous and liquid effluents at materials facilities
ES 114-4 Guidelines for Ground-Water Monitoring at In Situ Uranium Solution Mines	◙ Describes methods acceptable to the NRC staff for groundwater monitoring at in-situ uranium solution mines

Documentation

Documentation that addresses the monitoring plans of fuel cycle facilities can be found in preconstruction license applications, environmental reports that are required by NEPA, the

site/facility operating license, the decommissioning plan, and semiannual reports generated for license renewal applications.

The NRC reviews monitoring reports and conducts inspections at fuel cycle facilities to ensure that all applicable standards are being maintained. Any violations or deviations are noted, and concerns requiring management attention are identified.

MONITORING PROGRAMS AT FUEL CYCLE FACILITIES

Monitoring programs and regulations ensure the protection of personnel, the public, and the environment from unnecessary exposure to facility/site contamination. These regulations, laws, and other legally binding documents provide assurance to the public that the site/facility is operating within the legal parameters established by the government.

Monitoring activities at fuel cycle facilities are divided into preoperational, operational, and decommissioning stages.

Preoperational

In order to obtain a license, normally an applicant must submit a separate document, "The Applicant's Environmental Report," which is the appropriate NEPA documentation. This document is developed and submitted in two stages: (1) preconstruction and (2) postconstruction. The NEPA assessment provides the context for all subsequent monitoring activities and additionally requires the following:

- All potential impacts associated with the construction and operation of the facility must be considered.
- A risk-oriented framework must be developed to address the potential impacts of releases of both radiological and nonradiological contaminants to the environment.

This framework is expressed as a pathway analysis (conceptual model) and relates sources of contamination to receptors through environmental pathways.

Since the facility is not actually in operation during preconstruction, there are no effluents or other releases to monitor. Therefore, the effluents (source terms) are hypothesized based on known facility operations. Models are used with appropriate meteorological and hydrological inputs to estimate concentrations in the environment.

Purposes of Preoperational Monitoring

Purposes of preoperational monitoring:

- Provides information necessary to assess the environmental impacts and risks associated with proposed facility construction and operation
- Characterizes the site hydrological, meteorological, and geohydrological conditions to determine the fate and transport of effluent releases to the environment and otherwise assess impacts from the proposed action

- Establishes background (i.e., preexisting) levels of radiation and concentrations of radiological and nonradiological contaminants in abiotic and biotic media for impact assessment and site decommissioning
- Establishes baseline ecological conditions (biota and their habitats) for impact assessment
- Predicts maximum potential annual radiation doses to the public resulting from effluent releases
- Predicts maximum radionuclide concentrations that may be present in biota
- Is utilized to design operational-phase effluent and environmental monitoring programs

Operational

During fuel cycle facilities' operational stage, radiological monitoring is conducted to provide data for license applications and the semiannual reports that are generated as part of the license application renewal process.

Purposes of Operational Monitoring

Purposes of operational monitoring:

- Assesses the environmental impact of radiological and nonradiological contaminants in effluents, including estimates of the potential annual radiation doses to the public
- Demonstrates compliance with the regulations for concentrations of radiological contaminants in effluent releases in 10 CFR 20
- Characterizes effluent releases of radiological and nonradiological contaminants sufficient to quantify source terms for estimating concentrations in environmental media
- Assesses the adequacy and performance of effluent controls
- Is used to conduct environmental monitoring of biotic and abiotic media for levels of radiation and concentrations of radiological and nonradiological contaminants
- Is used to conduct ecological monitoring as required to provide ongoing assessment of environmental impacts
- Evaluates attainment of ALARA requirements
- Provides warning of accidental releases
- Provides support for license renewal applications

Decommissioning

Decommissioning is a required process for termination of a facility license and release of the facility for unrestricted use. 10 CFR 20, 30, 40, and 70 prescribe specific criteria for decommissioning nuclear facilities. If a licensee does not renew a license, then activities required for decommissioning must be performed on or before the expiration date of the license. The licensee must request in writing that the license be terminated.

This request is usually accompanied by a written decommissioning plan. The plan describes how the licensee will conduct a radiation survey and/or evaluation of the premises where licensed activities were carried out. It also provides guidance in submitting a report to the NRC of the final survey or other information to sufficiently demonstrate that the premises are suitable for release for unrestricted use.

Purposes of Monitoring for Decommissioning

Purposes of monitoring for decommissioning:

- Characterizes the nature and extent of residual radiological and nonradiological contamination
- Establishes acceptable levels of residual contamination in situations involving multiple contaminants
- Demonstrates that residual contamination at the plant and site has been remediated to the extent necessary to meet criteria for release for unrestricted use (through implementation of a planned final radiation survey)
- Describes background radioactivity and nonradiological contamination in media of concern
- Estimates volumes of, and contaminant concentrations in, wastes generated during decommissioning, including mixed wastes

The ultimate goal of the decommissioning process is to ensure that future uses of any licensed facility will not result in individuals being exposed to unacceptable levels of radiation and/or radioactive materials.

Sampling and measurement are the primary means for identification of types, quantities, and locations of contamination. They are also the methods used to verify whether the remediation was successful.

The NRC reviews and evaluates the information provided by the licensee, performs independent confirmation of site conditions, as appropriate, and makes a determination as to termination of the license.

MODULE 8.0: SAMPLING AND MEASUREMENT PRACTICES

ENVIRONMENTAL SAMPLING AND MEASUREMENT

The need for environmental sampling and measurement should be evaluated by pathway analysis for each radionuclide effluent stream. This evaluation should be documented in "The Applicant's Environmental Report." The site environmental monitoring plan should take into account actual emissions; if they exceed the criteria stated, then an analysis of the emission should be conducted.

The site/facility environmental monitoring program focuses on collecting data that will be used to evaluate any changes in environmental conditions that are a result of site/facility operations. Samples of air, surface water and groundwater, soil sediment, vegetation, aquatic plants and animals, and components in the food chain are withdrawn and analyzed for concentrations of radionuclides and other nonradiological contaminants that are a result of the fuel cycle site/facility.

Provisions should exist in the site emergency plan for environmental monitoring during emergencies. Provisions should be made for detection and quantification of unplanned releases of radionuclides to the environment.

Objectives of the Monitoring Plan

Overall plans and objectives should be established for a site/facility environmental monitoring plan. This plan should:

- Comply with all environmental quality standards and public exposure limits
- Develop background levels and site contribution of radioactive materials in the environment
- Determine the effectiveness of the effluent treatment program
- Determine the validity and effectiveness of models used to predict the concentration of pollutants in the environment
- Determine long-term buildup and prediction of environmental trends from site-released radioactive material
- Detect and quantify unplanned releases

Self-Check Questions 8-8

INSTRUCTIONS: Match the regulatory basis document in column A with its description in column B. Answers are located in the Trainee Guide.

Column A Document	Column B Description
A. National Environmental Policy Act	1.____ Establishes procedures and criteria for issuance of licenses to receive title to, own, acquire, deliver, receive, possess, and initially transfer special nuclear material.
B. Title 10 CFR Part 20 Standards for Protection Against Radiation	2.____ Requires federal government agencies to prepare a detailed statement on the environmental effects of proposed federal actions significantly affecting the quality of the human environment.
C. Title 10 CFR Part 70 Domestic Licensing of Special Nuclear Material	3.____ Establishes procedural requirements, generally applicable NRC health and safety standards, technical safety requirements, and safeguards and security requirements specific to gaseous diffusion plants.
D. Title 10 CFR Part 76 Certification of Gaseous Diffusion Plants	4.____ Establishes standards for protection against radiation hazards arising from activities under licenses issued by the NRC.

5. List two purposes of the preoperational monitoring stage.

6. List two purposes of the operational monitoring stage.

7. List two purposes of the decommissioning monitoring stage

8. What is the ultimate goal of the decommissioning process?

You have completed this section.
Please check off your progress on the tracking form.
Go to the next section.

Learning Objective

When you finish this section, you will be able to:

8.1.13 Identify the purpose of the Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM).

MULTI-AGENCY RADIATION SURVEY AND SITE INVESTIGATION MANUAL (MARSSIM)

Purpose and Scope

The Multi-Agency Radiation Survey and Site Investigation Manual provides detailed guidance for planning, implementing, and evaluating environmental and facility radiological surveys conducted to demonstrate compliance with a dose- or risk-based regulation. The MARSSIM guidance focuses on the demonstration of compliance during the final status survey following scoping, characterization, and any remedial actions.

Its objective is to describe a consistent approach for planning, performing, and assessing building surface and surface soil final status surveys to meet established dose- or risk-based release criteria, while at the same time encouraging an effective use of resources.

MARSSIM is a multiagency consensus document that was developed collaboratively by four federal agencies having authority and control of radioactive materials: Department of Defense (DOD), Department of Energy (DOE), Environmental Protection Agency (EPA), and the Nuclear Regulatory Commission (NRC).

Use of the Manual

Potential users of MARSSIM include federal, state, and local government agencies having authority for control of radioactive environmental contamination; their contractors; and other parties, such as organizations with licensed authority to possess and use radioactive materials. It is intended for a technical audience having knowledge of radiation health physics and an understanding of statistics as well as experience with the practical application of radiation protection.

Self-Check Questions 8-9

NSTRUCTIONS: Complete the following question. The answer is located in the answer key section of the Trainee Guide.

1. What is the purpose of the Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)?

It's time to schedule a progress meeting with your administrator. Review the progress meeting form on the next page. In Part III, as a Regulator, write your specific questions to discuss with the administrator.

PROGRESS REVIEW MEETING FORM

Date Scheduled: _____Location:_____

I. The following suggested items should be discussed with the administrator as to how they pertain to your current position:

- Sampling and measurement program
- Destructive and nondestructive measurements
- Sources of error in measurement process
- Sampling liquids in-process
- Liquid effluent/discharges
- Environmental water sampling and devices
- Sampling gases, equipment, and analysis
- Stack sampling
- Format for reporting effluent data
- Sampling solids
- Soil sampling
- Sampling and measurement of biota, vegetation, and food chain
- Sample collection and handling
- Sampling and measurement for maintaining nuclear criticality safety
- Regulatory guidance
- Preoperational, operational, and decommissioning monitoring
- MARSSIM

II. Use the space below to take notes during your meeting.

III. As a Regulator:

- Tell me about destructive and nondestructive measurements used at fuel cycle facilities.
- What problems have been encountered when sampling liquids in-process? Gases? Solids?
- How can I ensure that the "regulatee" is getting a representative sample?
- Show me some reports concerning effluent data and explain the significance of the data.
- Tell me about the MARSSIM document and how it is used by the NRC.

Use the space below to write your specific questions.

IV. Further assignments? If yes, please note and complete. If no, initial completion of progress meeting on tracking form.

As recommended by the administrator:

- Read one or more Title 10 CFR documents listed in Table 8-8.
- Read one or more Regulatory Guides listed in Table 8-9.
- Read select chapter(s) of MARRISM

MODULE 8.0: SAMPLING AND MEASUREMENT PRACTICES

Ensure that you and your administrator have dated and initialed your progress on your tracking form for this module.
Go to the module summary.

MODULE SUMMARY

Key Points:

- The NRC requires fuel cycle facilities to establish and maintain a sampling and measurement program. Regulatory objectives are:
  - Protect the health and safety of the public (including plant workers) and the environment from radiological and certain chemical hazards.
  - Promote the common defense and security at facilities that process special nuclear material by safeguarding the material from loss, theft, or diversion.
- Sampling involves extracting a representative portion of a material for subsequent measurement. Measurement involves quantifying the mass, volume/density, chemical composition, and/or another relevant property.
- Sampling and measurement activities are conducted to:
  - Establish and maintain control and accountability on all nuclear materials onsite
  - Verify adherence to product specifications and monitor process contaminants
  - Verify compliance with relevant federal, state, or local requirements
  - Maintain nuclear criticality safety
  - Quantify radiation exposures to plant workers and members of the public
  - Quantify impact of radiological and certain chemical hazards on the environment
- Measurements are necessary for facilities to know the
  - Amount of material received
  - Amount of material shipped
  - Amount of material discarded
  - Amount of material on inventory
- Measured material amounts are essential for resolving cases of loss of theft, or even allegations of loss or theft.
- Sampling and measurement activities also include:

- In-process monitoring
- Routine environmental monitoring
- Emergency monitoring

- Methods of sampling bulk nuclear material include:
  - Grab sample – a sample representative of a specific location at a given point in time, which is meaningful only if the material sampled is homogeneous
  - Continuous sample – repetitive, sequential collection of samples at intervals
  - Composite sample – a number of samples combined into a single sample
- Potential sources of sampling error include:
  - Failure to obtain homogeneity in the material being sampled
  - Inadequate protection of the sample while it is awaiting analysis
- Measurements can include both destructive and nondestructive assay (NDA).
  - Chemical measurement involves destroying a portion of the material.
  - NDA measurement does not destroy the sample mass or identity.
- Measurements include both volume and mass determination.
- The result of the measurement process will always differ to some extent from the true value. The difference between an observed or measured value and the true value is referred to as measurement error.
- Error is generally described in terms of two components, random and systematic:
  - How accurate? Systematic error is error that remains constant over a series of measurements. To control systematic error, measure known standards.
  - How precise? Random error occurs "randomly" over replicate measurements. To reduce random error, replicate measurements and use an average.
- Total measurement error (uncertainty) is the sum of random and systematic errors.
- In designing measurement quality assurance and quality control protocols, emphasis should be placed on those sources that contribute the largest amounts of error.
- Data validation is essential to maintaining the integrity of measurements. The validation process includes:
  - Acceptance criteria
  - Engineering studies of sampling/measurement systems and sources of error
  - Measurement of standards
  - Written procedures and procedural compliance

- Measurement error estimates
- Data review
- Records management

- A system for records management is essential. Such a system must address:
  - Records control
  - Archiving and indexing
  - Records accessibility
  - Storage, preservation, and safekeeping
  - Final disposition
- Decommissioning and Remediation:
  - A decommissioning process is required for termination of a facility license and release of the facility for unrestricted use.
  - Sampling and measurement are conducted to identify types, quantities, and locations of contamination.
  - A facility must know what materials it has before it can make decisions for decommissioning.
  - Sampling and measurement are also used to verify whether remediation was successful.

Congratulations! You are ready to go to the next assigned module.