10 FIELD AND SAMPLING ISSUES THAT AFFECT LABORATORY MEASUREMENTS

Part A: Generic Issues

10.1 Introduction

This chapter provides guidance to project managers, planners, laboratory personnel, and the radioanalytical specialists tasked with developing a field sampling plan. It emphasizes those activities conducted at the time of sample collection and other activities conducted after sample collection that could affect subsequent laboratory analyses.

A field sampling plan should provide comprehensive guidance for collecting, preparing, preserving, shipping, and tracking field samples and recording field data. The principal objective of a well-designed sampling plan is to provide representative samples of the proper size for analysis. Critical to the sampling plan are outputs of the systematic planning process, which commonly define the Analytical Protocol Specifications (APSs) and the measurement quality objectives (MQOs) that must be met. While comprehensive discussions on actual field sample collection and sampling strategies are beyond the scope of MARLAP, specific aspects of sample collection methods and the physical preparation and preservation of samples warrant further discussion because they impact the analytical process and the data quality.

This chapter has two main parts. Part A identifies general elements of a field sampling plan and provides project planners with general guidance. Part B provides detailed, matrix-specific guidance and technical data for liquid, solid, airborne, and surface contaminants requiring field sampling. This information will assist project planners further in the development of standard operating procedures (SOPs) and training for field personnel engaged in preparation and preservation of field samples.

The need to specify sample collection methods, and to prepare and preserve field samples, is commonly dictated by one or more of the following:

- The systematic planning process that identifies the type, quality, and quantity of data needed to satisfy a decision process;
- The potential alteration of field samples by physical, chemical, and biological processes during the time between collection and

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analysis;

- Requirements specified by the analytical laboratory pertaining to sample analysis;
- Requirements of analytical methods; and
- Requirements of regulators (e.g., Department of Transportation).

10.1.1 The Need for Establishing Channels of Communication

To design an effective sampling plan, it is critical to obtain the input and recommendations of representatives of (1) the field sampling team, (2) the health physics professional staff, (3) the analytical laboratory, (4) statistical and data analysts, (5) quality assurance personnel, and (6) end-users of data.

Beyond the initial input that assist the project planners in the design of the sampling plan, it is equally important to maintain open channels of communication among key members of the project team throughout the process. For example, the analytical laboratory should be provided with contacts within the field sampling team to ensure that modifications, discrepancies, and changes are addressed and potential problems may be resolved in a timely manner.

Communication among project staff, field personnel, and the laboratory offer a means to coordinate activities, schedules, and sample receipt. Project planning documents generated from the systematic planning process, such as APSs and statements of work (SOWs), should be consulted, but they cannot address all details. Additional communication will be necessary to convey information about the number and type of samples the laboratory can expect at a certain time. Documentation with special instructions regarding the samples should be received before the samples arrive. This information notifies the laboratory of any health and safety concerns so that laboratory personnel can implement proper contamination management practices. Health and safety concerns may affect analytical procedures, sample disposition, etc. The analytical laboratory should have an initial understanding about the relative number of samples that will be received and the types of analyses that are expected for specific samples. Furthermore, advance communications allow laboratory staff to adjust to modifications, discrepancies, and changes.

10.1.2 Developing Field Documentation

The field organization must conduct its operations in such a manner as to provide reliable information that meets the data quality objectives (DQOs). To achieve this goal, all relevant procedures pertaining to sample collection and processing should be based on documented standard operating procedures that may include, but are not limited to, the following activities:

- Developing a technical basis for defining the size of individual samples;

- Selecting field equipment and instrumentation;
- Using proper sample containers and preservatives;
- Using consistent container labels and sample identification codes;
- Documenting field sample conditions and exceptions;
- Documenting sample location;
- Tracking, accountability, custody, and shipment forms;
- Legal accountability, such as chain-of-custody record, when required;
- Selecting samples for field quality control (QC) program;
- Decontaminating equipment and avoiding sample cross-contamination;
- Specifying sample packaging, radiological surveys of samples, shipping, and tracking; and
- Documenting the health and safety plan.

10.2 Field Sampling Plan: Non-Matrix-Specific Issues

10.2.1 Determination of Analytical Sample Size

When collecting environmental samples for radiochemical analysis, an important parameter for field personnel is the mass or volume of an individual sample that must be collected. The required minimum sample size is best determined through the collective input of project planners, field technicians, and laboratory personnel who must consider the likely range of the contaminant concentrations, the type of radiation emitted by constituents or analytes (alpha, beta, and gamma emitters), field logistics, and the radioanalytical methods that are to be employed. It is important to have a quantitative understanding of the relationship between sample size and project specific requirements in order for samples to yield useful data.

10.2.2 Field Equipment and Supply Needs

Before starting field sampling activities, all necessary equipment and supplies should be identified, checked for proper operation and availability, and—when appropriate—pre-assembled. Instrumentation and equipment needs will depend not only on the matrix to be sampled, but also on the accessibility of the matrix and the physical and chemical properties of radionuclide contaminants under investigation.

In addition to specialized field equipment and instrumentation, field sampling supplies commonly include, but are not limited to, the following:

- Sampling devices (e.g., trowel, hand auger, soil core sampler, submersible water pump, high volume air filter, etc.);

- Sampling preparation equipment (e.g., weighing scales, volume measuring devices, soil screening sieves, water filtering equipment, etc.);

- Sample preservation equipment and agents (e.g., refrigeration, ice, formaldehyde or acid additives);
- Personnel protective gear (e.g., respiratory protective devices, protective clothing such as gloves and booties, life-preservers, etc.);
- Proper writing utensils (e.g., permanent pens and markers);
- Field logbooks and field tracking forms;
- Maps, distance measuring equipment, global positioning systems, or other location-determining equipment;
- Field sampling flags or paint;
- Chain-of-custody (COC) forms;
- Sample tags, labels, and documents;
- Appropriately labeled sample containers;
- Shipment containers and packing materials that meet national and international shipping regulations (see Section 10.2.10);
- Shipment forms;
- Analysis request forms identifying the type of radioanalysis to be performed; and
- Items required by the health and safety plan (medical kit, etc.).

10.2.3 Selection of Sample Containers

There are several physical and chemical characteristics to consider when selecting a suitable container for shipping and storing samples. These include the container material and its size, configuration, and method for ensuring a proper seal.

10.2.3.1 Container Material

Sample containers must provide reasonable assurance of maintaining physical integrity (i.e., against breakage, rupture, or leakage) during handling, transport, and potentially long periods of storage. The most important factor to consider in container selection is the chemical

compatibility between container material and sample. Containers may be made from ordinary bottle glass, borosilicate glass (such as Pyrex® or Corex®), plastics (e.g., high-density polyethylene, HDPE), low-density polyethylene, polycarbonate, polyvinyl chloride (PVC), fluorinated ethylene or propylene (Teflon™), or polymethylpentene. For certain samples, the choice of containers may require metal construction or be limited to paper envelopes.

10.2.3.2 Container Opening and Closure

A suitable container also should be shaped appropriately for the purpose. For example, a wide-mouthed container will provide easier access for the introduction and withdrawal of sample material and eliminate spills or the need for additional tools or equipment (e.g., funnel) that may become a source of cross contamination among samples.

Equally important is the container's closure. As a rule, snap-on caps should not be considered for liquid samples because they do not ensure a proper seal. Even when screw caps are used, it is frequently prudent to protect against vibration by securing the cap with electrical or duct tape. A proper seal is important for air samples, such as radon samples. The container cap material, if different from the container material, must be equally inert with regard to sample constituents.

10.2.3.3 Sealing Containers

Tamper-proof seals offer an additional measure to ensure sample integrity. A simple example includes placing a narrow strip of paper over a bottle cover and then affixing this to the container with a wide strip of clear tape (EPA, 1987, Exhibit 5-6 provides examples of custody seals). The paper strip can be initialed and dated in the field to indicate the staff member who sealed the sample and the date of the seal. Individually sealing each sample with a custody seal with the collector's initials and the date the sample was sealed may be required by the project. The seal ensures legal defensibility and integrity of the sample at collection. Tamper-proof seals should only be applied once field processing and preservation steps are completed. Reopening this type of sealed container in the field might warrant using a new container or collecting another sample.

10.2.3.4 Precleaned and Extra Containers

The reuse of sample containers is discouraged because traces of radionuclides might persist from initial container use to subsequent use. The use of new containers for each collection removes doubts concerning radionuclides from previous sampling. New containers might also require cleaning (ASTM D5245) to remove any plasticizer used in production or to pretreat glass surfaces. Retaining extra empty containers from a new lot or a special batch of precleaned and treated containers can provide the laboratory container blanks for use as part of quality control. Extra containers are also useful for taking additional samples as needed during field collection and to replace broken or leaking containers.

10.2.4 Container Label and Sample Identification Code

Each sample can only be identified over the life of a study if a form of permanent identification is provided with or affixed to the container or available in sample log. The most useful form of identification utilizes a unique identifier for each sample. Such unique identification codes ensure the project's ability to track individual samples. The standard operating procedure (SOP) that addresses sample identification should describe the method to be used to assure that samples are properly identified and controlled in a consistent manner. Containers sometimes may be pre-labeled with identification numbers already in place.

Any identification recorded on a container or a label affixed to the container should remain with the container throughout sample processing and storage. The identification information should be written with a permanent marker—especially if the labels are exposed to liquids. Information can be recorded directly on the container or on plastic or paper tags securely fixed to the container. However, tags are more likely to become separated from containers than are properly secured labels.

Labels, tags, and bar codes should be durable enough so no information is lost or compromised during field work, sample transport, or laboratory processing. Transparent tape can be used to cover the label once it is completed. The tape protects the label, adds moisture resistance, prevents tampering with the sample information, and helps secure the label to the container.

The project manager needs to determine if a field-sample identification (ID) scheme may introduce bias into the analysis process, such as allowing the laboratory to become aware of trends or locations from the sample identification. This could influence their judgment about the anticipated result and thereby introduce actions on the part of laboratory personnel that they would not otherwise take (such as reanalyzing the sample). The project manager needs to determine the applicability of electronic field data recorders and the issue of electronic signatures for the project.

A unique identifier can include a code for a site, the sample location at the site, or a series of digits identifying the year and day of year (e.g., "1997-127" uses the Julian date, and "062296" describes a month, day, and year). Alternatively, a series of digits can be assigned sequentially by site, date, and laboratory destination. The use of compass headings and grid locations also provides additional unique information (e.g., "NW fence, sampled at grid points: A1 through C25, 072196, soil"). With this approach, samples arriving at a laboratory are then unique in two ways. First, each sample can be discriminated from materials collected at other sites. Second, if repeat samples are made at a single site, then subsequent samples from the same location are unique only by date. Labeling samples sequentially might not be appropriate for all studies. Bar coding may reduce transcription errors and should be evaluated for a specific project.

10.2.5 Field Data Documentation

All information pertinent to field sampling is documented in a log book or on a data form. The log book should be bound and the pages numbered consecutively, and forms should be page-numbered and dated. Where the same information is requested routinely, preprinted log books or data sheets will minimize the effort and will standardize the presentation of data. Even when standardized preprinted forms are used, all information recorded should be in indelible ink, with all entry errors crossed out with a single line and initialed. The color of ink used should be compatible with the need to copy that information. All entries should be dated and signed on the date of entry. Initials should be legible and traceable, so that it is clear who made the entry.

Whenever appropriate, log or data form entries should contain—but are not limited to—the following:

- Identification of Project Plan or Sampling Plan;
- Location of sampling (e.g., reference to grid location, maps, photographs, location in a room);
- Date and time of sample collection;
- Sample matrix (e.g., surface water, soil, sediment, sludge, etc.);
- Suspected radionuclide constituents;
- Sample-specific ID;
- Sample volume, weight, depth;
- Sample type (e.g., grab, composite);
- Sample preparation used (e.g., removal of extraneous matter);
- Sample preservation used;
- Requested analyses to be performed (e.g., gross beta/gamma, gamma spectroscopy for a specific radionuclide, radiochemical analysis);
- Sample destination, including name and address of analytical laboratory;
- Names of field people responsible for collecting sample;

- Physical and meteorological conditions at time of sample collection;
- Special handling or safety precautions;
- Results of field radiation measurements, including surveys of sample containers; and
- Signatures or initials of appropriate field personnel. When using initials, ensure that they can be uniquely identified with an individual.

Labels affixed to individual sample containers should contain key information that forms an abstract of log book data sheets. When this is not practical, a copy of individual sample data sheets may be included along with the appropriately ID-labeled sample.

10.2.6 Field Tracking, Custody, and Shipment Forms

A sample tracking procedure must be in place for all projects in order that the proper location and identification of samples is maintained throughout the process from collection through handling, preservation, storage, transfer to laboratory, and disposal. The term "tracking" means an accountability process that meets generally acceptable laboratory practices as described by accrediting bodies, but is less stringent than a formal chain-of-custody process. Tracking also develops a record of all individuals responsible for the custody and transfer of the samples. Chapter 4 (*Project Plan Documents*) discusses the process of tracking and accountability. Also, Chapter 11 (*Sample Receipt, Inspection, and Tracking*) discusses the laboratory process of tracking.

When transferring the possession of samples, the individuals relinquishing and the individuals receiving the samples should sign, date, and note the time on the form. A standardized form should be designed for recording tracking or formal chain-of-custody information related to tracking sample possession. An example of a COC form is shown in Figure 10.1. Additional information and examples of custody forms are illustrated by EPA (1987 and 1994). If samples are to be split and distributed to more than one analytical laboratory, multiple forms will be needed to accompany sample sets. The sample collector is responsible for initiating the sample tracking record. The following information is considered minimal for sample tracking:

- Name of project;
- Sampler's signature;
- Sample ID;
- Sample location
- Date and time sampled;
- Sample type;
- Preservatives;
- Number of containers;

- Analysis required;
- Signatures of persons relinquishing, receiving, and transporting the samples;
- Signature for laboratory receipt;
- Method of shipment or carrier and air bill when shipped or shipping manifest identification upon receipt; and
- Comments regarding the integrity of shipping container and individual samples.

10.2.7 Chain of Custody

The legal portion of the tracking and handling process that ensures legal defensibility from sample collection to data reporting has become relatively standardized and is referred to as the

CHAIN-OF-CUSTODY RECORD

FIELD IDENTIFICATION NUMBER	FIELD LOCATION	DATE	TIME	SAMPLED BY:					
				SAMPLE MATRIX			SEQ. No.	No. of Containers	Analysis Required
				Water	Soil	Other			
Relinquished by:			Date/Time /	Received by:					Date/Time /
Relinquished by:			Date/Time /	Received by:					Date/Time /
Relinquished by:			Date/Time /	Received by:					Date/Time /
Relinquished by:			Date/Time /	Received by:					Date/Time /
Relinquished by:			Date/Time /	Received by laboratory for field analysis:					Date/Time /
Method of Shipment:									
Distribution: Orig. - Accompany Shipment 1 Copy – Survey Coordinator Field Files									

FIGURE 10.1—Example of chain-of-custody record

COC process (APHA, 1998). Guidance is provided in ASTM D4840 and NIOSH (1983). The level of security required to maintain an adequate chain of custody is that necessary to establish a "reasonable probability" that the sample has not been tampered with. For court proceedings, the requirements are established in law. COC procedures are important in demonstrating sample control when litigation is involved. In many cases, federal, state or local agencies may require that COC be maintained for specific projects. COC is usually not required for samples that are generated and immediately tested within a facility or continuous (rather than discrete or integrated) samples that are subject to real- or near-real-time analysis (e.g., continuous screening).

When COC is required, the custody information is recorded on a COC form. Chain-of-custody documents vary by organization and by project. Communication between field and laboratory personnel is critical to the successful use of COC. Any error made on a custody form is crossed out with a single line and dated and initialed. Use of correction ink or obliteration of data is not acceptable. Inform the laboratory when COC is required before the samples are received (see Section 11.2.4, "Sample Chain-of-Custody," for further information). The COC documents are signed by personnel who collect the samples. A COC record accompanies the shipment and one or more copies are distributed to the project coordinator or other office(s) where field and laboratory records are maintained.

10.2.8 Field Quality Control

A project plan should have been developed to ensure that all data are accurate and that decisions based on these data are technically sound and defensible. The implementation of a project plan requires QC procedures. QC procedures, therefore, represent specific tools for measuring the degree to which quality assurance objectives are met. Field QC measures are discussed comprehensively in ASTM D5283.

While some types of QC samples are used to assess analytical process, field QC samples are used to assess the actual sampling process. The type and frequency of these field QC samples must be specified by the project planning process along with being included in the project planning documents and identified in the sampling plan. Definitions for certain types of field QC samples can be found in ASTM D5283 and MARSSIM (2000).

10.2.9 Decontamination of Field Equipment

Sampling SOPs must describe the recommended procedure for cleaning field equipment before and during the sample collection process, as well as any pretreatment of sample containers. The SOPs should include the cleaning materials and solvents used, the purity of rinsing solution or water, the order of washing and rinsing, associated personnel safety precautions, and the disposal of cleaning agents.

Detailed procedures for the decontamination of field equipment used in the sampling of low-

activity soils, soil gas, sludges, surface water, and ground water are given in ASTM D5608.

10.2.10 Packing and Shipping

The final responsibility of field sampling personnel is to prepare and package samples properly for transport or shipment by a commercial carrier. All applicable state and federal shipping requirements, discussed later in this section, must be followed. When samples must be shipped by commercial carrier or the U.S. Postal Service, containers must be designed to protect samples against crushing forces, impacts, and severe temperature fluctuations. Within each shipping container, the cushioning material (sawdust, rubber, polystyrene, urethane foam, or material with similar resiliency) should encase each sample completely. The cushioning between the samples and walls of the shipping containers should have a minimum thickness of 2.5 cm. A minimum thickness of five centimeters should be provided on the container floor.

Samples should also be protected from the potentially adverse impacts of temperature fluctuations. When appropriate, protection from freezing, thawing, sublimation, evaporation, or extreme temperature variation may require that the entire interior surface of the shipping container be lined with an adequate layer of insulation. In many instances, the insulating material also may serve as the cushioning material.

The requirements for container security, cushioning, and insulation apply regardless of container material. For smaller volume and low-weight samples, properly lined containers constructed from laminated fiberboard, plastic, or reinforced cardboard outer walls also may be used.

When samples are shipped as liquids in glass or other breakable sample containers, additional packaging precautions may have to be taken. Additional protection is obtained when sample containers are shipped in nested containers, in which several smaller containers (i.e., inner containers) are packed inside a second larger container (i.e., the outer pack or overpack). To contain any spills of sample material within the shipping container, it is advisable either to wrap individual samples or to line the shipping container with absorbent material, such as asbestos-free vermiculite or pearlite.

For proper packaging of liquid samples, additional guidance has been given by EPA (1987) and includes the following:

- All sample bottles are taped closed;
- Each sample bottle is placed in a plastic bag and the bag is sealed;
- Each sample bottle may be placed in a separate metal can filled with vermiculite or other packing material, and the lid taped to the can;

- The cans are placed upright in a cooler that has its drain plug taped closed, inside and out, and lined with a plastic bag; and

- The cooler is filled with packing material—"bubble wrap" or cardboard separators may be used—and closed with sealing tape.

Field screening measurements are made for compliance with U.S. Department of Transportation regulations, 49 CFR Parts 170 through 189, as well as compliance with the laboratory's license from the U.S. Nuclear Regulatory Commission (NRC; 10 CFR Part 71) and Agreement State (if applicable). International requirements may also apply. See the International Air Transport Association's Dangerous Goods Regulations for additional guidance. These regulations not only set contamination and radiation levels for shipping containers, but also describe the types of containers and associated materials that are to be used based on the total activity and quantity of materials shipped. When the samples are screened in the field with survey instrumentation, the results should be provided to the laboratory. This information should also state the distance used from the probe to the packing container wall. Measurements normally are made in contact or at one meter. The readings in contact are most appropriate for laboratory use. The screening measurements in the field are mainly for compliance with transportation requirements and are usually in units of exposure. Laboratory license requirements are usually by isotope and activity. Project planning and communication are essential to ensure that a specific set of samples can be transported, received, and analyzed safely while complying with applicable rules and regulations.

The external surface of each shipping container must be labeled clearly, contain information regarding the sender and receiver, and should include the respective name and telephone number of a contact. When required, proper handling instructions and precautions should be clearly marked on shipping containers. Copies of instructions, shipping manifest or container inventory, chain of custody, and any other paperwork that are enclosed within a shipping container should be safeguarded by placing documents within a sealed protected envelope.

10.2.11 Worker Health and Safety Plan

In some cases, field samples will be collected where hazardous agents or site conditions might pose health and safety considerations for field personnel. These can include chemical, biological, and radiological agents, as well as common industrial hazards associated with machinery, noise levels, and heat stress. The health and safety plan established in the planning process should be followed. For the U.S. Department of Defense, these plans may include imminent threats to life, such as unexploded ordnance, land mines, hostile forces, chemical agents, etc. A few of the hazards particular to field sampling are discussed in the following sections, but these should not be construed as a comprehensive occupational health and safety program. The Occupational Safety and Health Administration's (OSHA) regulations governing laboratory chemical hygiene plans are located at 29 CFR 1910.1450. These requirements should apply as well to field sampling.

10.2.11.1 Physical Hazards

MECHANICAL EQUIPMENT

Personnel working with hand-held tools (e.g., sledge hammers used for near-surface coring) or power tools and equipment are subject to a variety of hazards. For example, personnel drilling monitoring wells are exposed to a variety of potential mechanical hazards, including moving machinery, high-pressure lines (e.g., hydraulic lines), falling objects, drilling through underground utilities, flying machinery parts, and unsafe walking and working surfaces. The consequences of accidents involving these physical hazards can range from minor to fatal injury.

At a minimum, workers should be required to wear protective clothing, which includes hard hats, gloves, safety glasses, coveralls (as an option) and steel-toed safety shoes. Workers required to climb (e.g., ladders, drilling masts) must be trained according to OSHA standards in the proper use of devices to prevent falls.

For sampling operations that require drilling, open boreholes and wells must be covered or secured when unattended, including during crew breaks.

ELECTRICAL HAZARDS

Electric power often is supplied by gasoline or diesel engine generators. Working conditions may be wet, and electrical shock with possibly fatal consequences may occur. In addition, drilling operations may encounter overhead or buried electrical utilities, potentially resulting in exposure to very high voltages, which could be fatal or initiate fires.

All electrical systems used during field operations should be checked for proper grounding during the initial installation. Temporary electrical power provided to the drill site shall be protected by ground-fault circuit interrupters.

NOISE HAZARDS

Power equipment is capable of producing sound levels in excess of 85 dB(A), the eight-hour threshold limit value recommended by the American Conference of Governmental Industrial Hygienists. Exposure to noise levels in excess of 85 dB(A) for long periods of time can cause irreversible hearing loss. If noise levels exceed 85dB(A), a controlled area must be maintained at this distance with a posting at each entrance to the controlled area to read:

CAUTION NOISE HAZARD Hearing Protection Required Beyond This Point

HEAT STRESS

The use of protective clothing during summer months significantly increases the potential for personnel to experience heat stress. Adverse effects from heat stress include heat cramps, dehydration, skin rash, heat edema, heat exhaustion, heat stroke, or death. When heat stress conditions exist, the following ought to be available:

- A cool and shaded rest area;
- Regular rest breaks;
- An adequate supply of drinking water; and
- Cotton coveralls rather than impermeable Tyvek® coveralls.

CHEMICAL AND RADIOLOGICAL HAZARDS

The health and safety plan should contain information about a site's potential radionuclides and hazards that might be encountered during implementation of field sampling and survey procedures. All field personnel should read the health and safety plan and acknowledge an understanding of the radiological hazards associated with a site. Site specific training must be provided that addresses the chemical and radiological hazards likely to be associated with a site. Field procedures should include either information relating to these hazards or should reference appropriate sections of the health and safety plan. References related to the use of protective clothing are given in EPA (1987), DOE (1987, Appendix J), and in 29 CFR 1910, Subpart I.

When procuring environmental solid and liquid samples, unusual characteristics such as color, suspended material, or number of phases and unusual odors should be noted and a description should be provided to the on-site safety officer as well as the analytical laboratory. Additional information concerning field methods for rapid screening of hazardous materials is presented in EPA (1987). This source primarily addresses the appearance and presence of organic compounds that might be present on occasions when one is collecting materials to detect radioactivity. Checking samples for chemical or radiological hazards can be as simple as visual inspection or using a hand-held radiation meter to detect radiation levels. Adjustments to laboratory procedures, particularly those involving sample handling and preparation, can only be made when pertinent field information is recorded and relayed to the project planner and to the laboratory. In some cases, a laboratory might not have clearance to receive certain types of samples (such as explosives or chemical agents) because of their content, and it will be necessary to divert these samples to an alternate laboratory. It might be necessary to reduce the volume sampled in order to meet shipping regulations if high concentrations of radioactivity are present in the samples. In some cases, the activity of one radionuclide might be much higher than others in the same sample. Adjustments made on the basis of the radionuclide of higher activity might result in collection of too little of another radionuclide to provide adequate detection and thus prevent identification of these radionuclides because of their relatively low minimum detectable concentrations. These situations should be considered during planning and documented in the

appropriate sampling plan document.

10.2.11.2 Biohazards

Precautions should be taken when handling unknown samples in the field. Some examples are wearing gloves, coveralls or disposable garments, plastic booties, dust masks or other respiratory protection. Some biohazards may be snakes, ticks, spiders, and rodents (Hanta virus). Prevention of potential exposure is the goal of a safety program. The type of protective equipment in the field should be discussed in the planning process and specified in the appropriate plan document. Since there are many specifics that are site dependent, it is difficult to create a comprehensive list. But the information is discussed to provide an awareness and starting point for additional discussion.

PERSONNEL TRAINING AND QUALIFICATION

All field operations that could lead to injury for sample collectors should be performed by personnel trained to documented procedures. When sampling is conducted in radiologically controlled areas (RCAs) as defined in regulatory standards (i.e., 10 CFR 20, 10 CFR 835). Formal training and qualification of field personnel may be required.

Training may require both classroom and practical applications in order to familiarize personnel with the basic theory of radiation and radioactivity and the basic rules for minimizing external exposures through time, distance, shielding, and avoidance of internal exposure (by complying with rules regarding smoking, drinking, eating, and washing of hands). Other topics to cover include common routes of exposure (e.g., inhalation, ingestion, skin contact); proper use of equipment and the safe handling of samples; proper use of safety equipment such as protective clothing, respirators, portable shielding, etc.

Guidance for the training and qualification of workers handling radioactive material has been issued by the Nuclear Regulatory Commission (see appropriate NRC NUREGs and Regulatory Guides on training of radiation workers), Department of Energy (1994a–d), and the Institute of Nuclear Power Operations (INPO 88-010). These and other documents should be consulted for the purpose of training and qualifying field personnel.

PERSONNEL MONITORING AND BIOASSAY SAMPLING

When conditions dictate the need for personnel monitoring, various methods are commonly employed to assess external and internal exposure that might have resulted from the inhalation or ingestion of a radionuclide.

Thermoluminescent dosimeters, film badges, or other personnel dosimeters may be used to monitor and document a worker's external exposures to the whole body or extremities. For

internal exposures, assessment of dose may be based on: (1) air monitoring of the work area or the worker's breathing zone; (2) *in vivo* bioassay (whole-body counting); or (3) *in vitro* bioassays that normally involve urinalysis but also may include fecal analysis and nasal smears. For *in vitro* bioassays (i.e., urine or fecal), the standard method involves a 24-hour sample collection in a sealable container. Samples may be kept under refrigeration until laboratory analysis can be performed to retard bacterial action. (Bioassay sample collection is normally not performed in the "field.")

The following guidance documents may be used for personnel monitoring and the collection and preservation of bioassay samples:

- ANSI/ANS HPS N13.30 (1996), Performance Criteria for Radiobioassay;
- ANSI/ANS HPS N13.14 (1994), Internal Dosimetry Programs for Tritium Exposure—Minimum Requirements;
- ANSI/ANS HPS 13.22 (1995), Bioassay Programs for Uranium;
- ANSI/ANS HPS 13.42 (1997), Internal Dosimetry for Mixed Fission Activation Products;
- DOE Implementation Guide, Internal Dosimetry Program, G-10 CFR 835/C1—Rev. 1 Dec. 1994a;
- DOE Implementation Guide, External Dosimetry Program, G-10 CFR 835/C2—Rev. 1 Dec. 1994b;
- DOE Implementation Guide, Workplace Air Monitoring, G-10 CFR 835/E2-Rev. 1 Dec. 1994c;
- DOE Radiological Control Manual, DOE/EH-0256T, Rev. 1, 1994d;
- NRC Regulatory Guide 8.9, Acceptable Concepts, Models, Equations, and Assumptions for a Bioassay Program (September 1993);
- NRC Regulatory Guide 8.11, Applications of Bioassay for Uranium (Revision 1, July 1993);
- NRC Regulatory Guide 8.20, Applications of Bioassay for ^{125}I and ^{131}I (June 1974);
- NRC Regulatory Guide 8.22, Bioassays at Uranium Mills (Revision 1, August 1988);
- NRC Regulatory Guide 8.26, Applications of Bioassay for Fission and Activation Products (September 1980);
- NRC Regulatory Guide 8.32, Criteria for Establishing a Tritium Bioassay Program (July 1988);
- NCRP (1987), Use of Bioassay Procedures for Assessment of Internal Radionuclides Deposition; and
- INPO (1988), Guidelines for Radiological Protection at Nuclear Power Stations.

Part B: Matrix-Specific Issues That Impact Field Sample Collection, Processing, and Preservation

Field processing should be planned in advance so that all necessary materials are available during field work. Preparing checklists of processing equipment, instruments, and expendable

materials—exemplified in part by lists accompanying sampling procedures described by EPA (1994)—helps this planning effort and serves to organize field methods. Field personnel who communicate problems should prevent loss of time, effort, and improper sample collection, as well as documents exactly what equipment, instruments, etc. were used.

The initial steps taken in the field frequently are critical to laboratory analysis performed hours, days, or even weeks after a sample is obtained. Various sample preparation steps may be required before samples are packaged and shipped for laboratory analysis. The need for sample processing and preservation is commonly determined by the sample matrix, the DQOs of the analysis, the nature of the radionuclide, and the analytical method.

The goal of sample preservation is to maintain the integrity of the sample between the time the sample is collected and the time it is analyzed, thus assuring that the analysis is performed on a sample representative of the matrix collected. Sample preservation should limit biological and chemical actions that might alter the concentration or physical state of the radionuclide constituents or analytes. For example, cations at very low concentrations can be lost from solution (e.g., cesium can exchange with potassium in the glass container, and radionuclides can be absorbed by algae or slime growths in samples or containers that remain in the field for extended periods). Requirements for sample preservation should be determined during project planning when analytical protocols are selected. Sample preservation in the field typically follows or accompanies processing activities. Sample preservatives may be added to sample collection containers before they are sent to the field.

This section provides matrix-specific guidance that focuses on the preparation and processing of field samples. In order to assist project planners in developing a sampling plan, a limited discussion is also provided that describes matrix-specific methods commonly employed for the collection of field samples. Guidance is presented for only the most common materials or environmental media, which are generically classified as liquids, solids, and air. In some instances, a solid material to be analyzed involves particulate matter filtered from a liquid or air suspension. Because filter media can affect analytical protocols, a separate discussion is provided that addresses sample materials contained on filter materials, including surface contamination associated with wipe samples.

10.3 Liquid Samples

Liquid samples typically are classified as aqueous, nonaqueous, or mixtures. Aqueous samples requiring analysis are likely to represent surface water, ground water, drinking water, precipitation, tanks and lagoons, and runoff. Nonaqueous liquids may include a variety of solvents, oils and other organic liquids. Mixtures of liquids represent a combination of aqueous and nonaqueous liquids or a solid suspended in either aqueous and nonaqueous liquids. Standardized water sampling procedures are described in numerous documents (APHA, 1998;

EPA, 1985; EPA, 1987; DOE, 1997; ASTM D3370). Important decisions include the choice of instrument or tool used to obtain the sample, the sample container material, the need for sample filtration, and the use of sample preservatives.

10.3.1 Liquid Sampling Methods

The effect of the sample collection process on the sample integrity needs to be understood and managed. Two examples are dissolved gases and cross-contamination. It may be necessary to minimize dissolved oxygen and carbon dioxide, which can cause some dissolved metals to undergo reaction or precipitation.

Sampling is discussed in NAVSEA (1997) and USACE (1995). The latter reference has been superseded, but the revision does not include sampling. The sampling references listed in USACE (1995) are:

- U.S. Environmental Protection Agency (EPA). 1984. *Characterization of Hazardous Waste Sites—A Method Manual, Vol. II, Available Sampling Methods*, Second Edition, EPA 600-4-84-076.

- U.S. Environmental Protection Agency (EPA). 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater*, EPA 600-4-82-029.

- U.S. Environmental Protection Agency (EPA). 1986. *Compendium of Methods for Determination of Superfund Field Operation Methods*, EPA 600-4-87/006.

- U.S. Environmental Protection Agency (EPA). 1987. *A Compendium of Methods for Determination of Superfund Field Operation Methods*, EPA 540-P-87-001a, OSWER Directive 9355.0-14.

- U.S. Department of the Interior (DOI). 1980. *National Handbook of Recommended Methods for Water for Water-Data Acquisition*, Volume I and II.

10.3.2 Liquid Sample Preparation: Filtration

Filtration of a water sample may be a key analytical planning issue and is discussed in Section 3.4.3, “Filters and Wipes.” A decision needs to be made during project planning whether or not to filter the sample in the field. Filtration of water or other liquids may be required to determine contaminant concentrations in solubilized form, suspended particulates, or sediment. The method of filtration will depend on the required sample volume, the amount and size of suspended particulates, and the availability of portable equipment and resources (e.g., electricity).

The potential need to filter a water sample principally depends on the source of water and the

objectives of the project investigation. If, for example, the intent is to assess human exposure from ingestion of drinking water "at-the-spigot," unfiltered tap water samples are likely to be required. Conversely, filtration may be required for water taken from an unlined field monitor well that is likely to contain significant amounts of particulate matter. These solids are of little relevance but may interfere with radioanalytical protocols (e.g., sample absorption may occur during gross alpha or beta counting where the analytical procedure involves the simple evaporation of a water aliquant on a planchet).

For remote sampling sites, sample processing may be restricted to gravity filtration that requires a minimum of equipment and resources. Drawing samples through filters by pressure or suction that is created by syringe, vacuum pump, or aspiration are alternative options. If filter papers or membranes capture materials that will be retained for analysis, they should be handled with clean rubber or plastic gloves, forceps, or other instruments to prevent sample contamination.

Each federal agency may have unique guidance to determine the need and process for filtering samples. One performance-based example is that of EPA, discussed in the next section. This guidance applies to either the field or laboratory filtration.

10.3.2.1 Example of Guidance for Ground-Water Sample Filtration

After considering whether or not to filter ground-water samples when analyzing for metals, the Environmental Engineering Committee of EPA's Science Advisory Board (EPA, 1997) recommended:

- Several factors could introduce errors in the sampling and analysis of ground water for metals or metallic radionuclides. Well construction, development, sampling, and field filtering are among the steps that could influence the metals measured in the ground-water samples. Field filtering is often a smaller source of variability and bias compared to these other factors. Therefore, the Agency should emphasize in its guidance the importance of proper well construction, development, purging, and water pumping rates so that the field filtering decisions can also be made accurately.

- Under ideal conditions, field-filtered ground-water samples should yield identical metals concentrations when compared to unfiltered samples. However, under non-ideal conditions, the sampling process may introduce geological materials into the sample and would require field filtration. Under such conditions, filtering to remove the geological artifacts has the potential of removing colloids (small particles that may have migrated as suspended materials that are mobile in the aquifer). Available scientific evidence indicates that when wells have been properly constructed, developed, and purged, and when the sample has been collected without stirring or agitating the aquifer materials (turbidity less than 5 nephelometric turbidity units, NTU), then field filtering should not be necessary. For Superfund site assessments, the low-flow sampling technique without filtration is the preferred sampling

approach for subsequent metal analysis when well construction, well maintenance, and hydrogeological conditions such as flow rate allow. Under such conditions, the collected samples should be representative of the dissolved and particulate metals that are mobile in ground-water systems. The Agency's proposal to rely on low flow sampling and unfiltered samples is a conservative approach that favors false positives over false negatives.

- When the turbidity of the sample is high, the situation is different. In-line filtering provides samples that retain their chemical integrity. Therefore, field filtering of properly collected ground-water samples should be done when turbidity in the samples is higher than 5 NTU, even after slow pumping has been utilized to obtain the sample.

They acknowledged, however, that differences in the way wells are installed, their packing materials, and the techniques used to collect ground-water samples can lead to variability in analytical results between wells and between individual samples. Filtering a sample can be a way to remove suspended particles and some colloids that contain metals that would not normally be in the ground water if the material were not disturbed during sampling. Here, a colloid is defined as a particle that ranges in size from 0.003 to 10 μm (Puls et al., 1990; Puls and Powell, 1992). The literature indicates that colloids as large as 2 μm can be mobile in porous media (Puls and Powell, 1992). Saar (1997) presents a review of the industry practice of filtration of ground-water samples. For some sites with low hydraulic conductivity the presence of an excess of colloids presents numerous monitoring challenges and field filtration might be necessary.

The desire to disturb the aquifer as little as possible has led to the use of low-flow sampling of wells—low-flow purging and sampling occurs typically at 0.1 to 0.3 L/min (Saar, 1997). The low-flow technique maximizes representativeness by (EPA, 1997):

- Minimizing disturbances that might suspend geochemical materials that are not usually mobile;

- Minimizing disturbances that might expose new reactive sites that could result in leaching or adsorption of inorganic constituents of ground water;

- Minimizing exposure of the ground water to the atmosphere or negative pressures, ensuring that the rate of purging and sampling does not remove ground water from the well at a rate much greater than the natural ground-water influx; and

- Monitoring indicator parameters to identify when stagnant waters have been purged and the optimum time for sample collection.

In summary, based on the ability of the low-flow sampling technique to collect representative samples, EPA suggests that filtering of ground-water samples prior to metals analysis is usually not required (EPA, 1997).

10.3.2.2 Filters

The removal of suspended particles is commonly achieved by filtration. When filtration is required, it should be done in the field or as soon as practicable. Field filtration permits acid preservatives to be added soon after collection, which minimizes the adsorption of soluble contaminants on the container walls and avoids the dissolution of particulate matter which may not be part of the sample to be analyzed.

An arbitrary size of 0.45 μm has gained acceptance as the boundary between soluble and insoluble matter (particularly for water in power plant boilers (ASTM D6301). It is the filter pore size that is commonly recommended by laboratory protocols. Material that may be present in colloidal form (a second phase in a liquid that is not in solution), can have particles that range from 0.001 to 2 μm. Such particles may be problematic since they may or may not be filterable (Maron and Lando, 1974). Thus, there can be no single standard for filter type or pore size, and every project should establish its own filtration protocol based upon its needs.

The fact that small particles pass through membrane filters has been recognized for some time (Kennedy et al., 1974). Conversely, as the filters clog, particles an order of magnitude smaller are retained by these filters (Sheldon and Sutcliffe, 1969). It should be noted, however, that manufacturers of filters usually specify only what will not pass through the filter; they make no claims concerning what actually does pass through the filter. Laxen and Chandler (1982) present a comprehensive discussion of some effects of different filter types. They refer to thin (5 to 10 μm) polycarbonate filters as "screen types," and thick (100 to 150 μm) cellulose nitrate and acetate filters as "depth type." The screen-type filters (e.g., polycarbonate) clog much more rapidly than the depth type (e.g., cellulose nitrate and acetate) filters. Once the filtration rate drops, particles that would normally pass through the filter are trapped in the material already retained. Also, filtering through screen-type filters may take considerable time and may require suction or pressure to accomplish in a reasonable time. Hence, the use of screen-type filters, because of their increased propensity to clog, generally is not recommended.

In addition to the difficulty of contending with clogging, Silva and Yee (1982) report adsorption of dissolved radionuclides on membrane filters. Although these drawbacks cannot be completely overcome, they are still less than the potential difficulties that arise from not filtering.

Finally, good laboratory practices must be used for field sampling. The most likely sources of contamination for the filters are improperly cleaned tubing and filter holders and handling the filters with contaminated fingers. Tubing and holders should be thoroughly cleaned and rinsed between samples and the entire system should be rinsed several times with the water to be sampled. Filters should be handled with clean rubber gloves.

10.3.3 Field Preservation of Liquid Samples

Sample degradation may occur between the time of collection and analysis due to microbial contaminants or chemical interactions. Although sample degradation cannot destroy or alter the radiological properties of a contaminant, it can alter the radionuclide's chemical properties and its potential distribution within a sample. For example, microbial processes are known to affect both the chemical state and the distribution of radioelements due to oxidation-reduction reactions, complexation and solubilization by metabolic compounds, bioaccumulation, biomylation, and production of gaseous substances such as CO_2, H_2, CH_4, and H_2S (Francis, 1985; Pignolet et al., 1989).

The selected field preservation method also should take into account compatibility with the radionuclides, analytical methods, analytical requirements, and container properties (see Section 10.2.3, "Selection of Sample Containers"). One example that illustrates compatibility with the analytical method is the addition of HCl to water samples as a preservative for gross alpha and gross beta analyses. The HCl will corrode stainless steel planchets used in the method. If laboratory personnel are aware of this, they can include steps to prevent the corrosion. Other preservation issues for liquid samples are discussed in Table 10.1 (page 10-25). Compatibility issues should be evaluated during the planning phase and included in the field sampling plan.

10.3.3.1 Sample Acidification

Acidification is the method of choice for preserving most types of water samples. The principal benefit of acidification is that it keeps many radionuclides in solution and minimizes their potential for removal by chemical and physical adsorption or by ion exchange. The mode by which a radionuclide is potentially removed from solution is strongly affected by the radionuclide and the container material. For example, studies conducted by Bernabee et al. (1980) and Milkey (1954) demonstrated that the removal of metal ions from solution is dominated by physical (i.e., van der Waals) adsorption. Milkey's conclusion is based on: (1) the observation that the loss of uranium, lead, and thorium ions from solution was significantly greater for containers made of polyethylene than of borosilicate glass; and (2) the fact that while adsorption by glass may potentially involve all three adsorption processes; with polyethylene plastic, there are no valence-type attractive forces or ions to exchange, and only physical van der Waals adsorption is possible.

Similar observations were reported by: (1) Dyck (1968), who compared long-term adsorption of silver ions by molded plastic to glass containers; (2) Jackson (1962), who showed that polyethylene containers absorbed about five times as much ^{90}Sr as glass containers at pH of about seven; and (3) Martin and Hylko (1987a; 1987b), who reported that greater than 50 percent of ^{99}Tc was adsorbed by polyethylene containers from non-acidified samples.

For sample acidification, either nitric or hydrochloric acid is commonly added until a pH of less than two (APHA, 1998, Table 7010.1; EPA, 1980, Method 900.0). Other guidance for sample

preservation by acidification is summarized below.

In instances of very low-activity samples where container adsorption poses a significant concern, but where acidification of the sample interferes with the radioanalytical method, the choice of sample container may be limited to glass or require alternative methods. For example, the use of acids as a preservative is not recommended for the analysis of tritium (^{3}H), carbon-14 (^{14}C), or radon in water, and precautions must be taken for the following reasons:

- For radon, sample preservation offers no benefit and is therefore not required for analytical accuracy. Adding acid also may cause the generation of CO_2 in the sample, which could purge radon gas.

- The addition of acid to a sample containing ^{14}C may result in the production of $^{14}CO_2$ and the loss of ^{14}C from the sample.

- Acid does not have a direct effect on tritium. However, it may affect the cocktail used in liquid scintillation analysis, or as with HCl, may add significant quench to the cocktail (see Section 15.5.3, "Liquid Scintillation").

Although acidification has been shown to effectively reduce the adsorption of technetium by polyethylene, technetium in the TcO_4^{-4} state has been observed to volatilize in strong acid solutions during evaporation while preparing water samples for gross beta analysis (NAS, 1960). To hasten evaporation, the planchet is commonly flamed. This dilemma can be resolved by either precoating planchets with a film of detergent prior to the addition of the acidified water sample or by passive evaporation of the acidified water sample that avoids the higher temperature associated with flaming (Blanchard et al., 1993).

10.3.3.2 Non-Acid Preservation Techniques

If a sample contains significant organics, or if contaminants under investigation react with acids that interfere with the radioanalytical methods, other methods of sample preparation should be considered.

REFRIGERATION AND FREEZING

The effect of refrigeration or freezing temperatures to arrest microbial activity is a fundamental concept. Temperatures near the freezing mark or below not only retard or block bacterial growth but arrest essentially all other metabolic activity. It should, however, be noted that most bacteria can survive even in extreme temperatures. (Indeed, if a suspension of bacterial cells is frozen rapidly with no appreciable formation of ice crystals, it can be kept at temperatures as low as -194 °C for indefinite periods of time with little loss of viability.)

The choice between refrigeration and freezing is dictated by the potential impacts of ice formation on sample constituents. Besides physical changes of organic constituents, the initial formation of ice crystals and the exclusion of any solutes may concentrate the solutes to the point of precipitation. Quick freezing methods that minimize ice crystal formation are beneficial for preserving some organic constituents. Quick freezing is commonly done by packing sealed samples in liquid nitrogen or dry ice. Care must be taken, however, to avoid container breakage due to sample volume expansion. An air space of a least 10 percent and a container made of plastic provide reasonable assurance for container integrity.

When refrigeration is employed, attempts should be made to avoid temperatures that could result in slow freezing and the formation of ice crystals. Optimum refrigeration temperatures for sample preservation at 4 ± 2 °C can be achieved by packing samples in ice or freeze packs within a thermally insulated leak-proof container (ASTM D3856; ASTM D3370).

PAPER PULP

The addition of paper pulp, with its adsorptive property and large surface area, can avoid the adsorption and loss of easily hydrolyzed radionuclides to the container wall over time (Bernabee et al., 1980). About two grams of finely ground paper pulp are added per liter of acidified sample at time of collection. The pH should be adjusted to one or less and vigorously shaken. The sample may be stored in this condition for an extended period of time. To prepare for analysis, the pulp is removed from solution by filtration and subjected to wet ashing using strong acids (Chapter 12, *Laboratory Sample Preparation*). This ashed solution is commonly added to the original filtrate to make a reconstituted sample solution.

The use of paper pulp and the need for wet ashing, however, pose problems for certain radioanalytical laboratory protocols and must therefore be thoroughly evaluated.

SULFITE

To prevent the loss of radioiodine from solution, sodium bisulfite ($NaHSO_3$), sodium thiosulfate ($Na_2S_2O_3$), or sodium metabisulfite ($Na_2S_2O_5$) may be used. These compounds are strong reducing agents and will convert volatile iodine (I_2) to nonvolatile iodine (I^-). If acid is also employed to preserve samples for analysis of other radionuclides, it is important to note that acid will counteract the effectiveness of the reducing agent. For this reason, samples collected for iodine analyses typically are collected and preserved in a separate container. It should also be noted that the reducing environment produced by the sulfite-type preservatives may convert iron, uranium, and other reducible ions or their compounds to a different oxidation state. The inadvertent change in oxidation state of other radionuclides will have an obvious adverse impact on radioanalytical measurements that require chemical separation. Section 14.9 has additional information on carriers and tracers.

OTHERS

Other methods that have been used to preserve liquid samples containing organics and biological materials include chemical preservatives (e.g., formaldehyde and methanol). Table 10.1 summarizes the advantages and disadvantages of these and previously described preservation methods.

TABLE 10.1—Summary of sample preservation techniques.

Preservation Technique	Advantages	Disadvantages
Addition of HNO_3	Reduces pH and inhibits plating of metals on container walls.	Strong oxidizer that might react with organic compounds, such as liquid scintillation cocktails. ^{14}C might be lost as $^{14}CO_2$.
Addition of HCl	Reduces pH and inhibits plating of metals on container walls. Chloride forms strong anionic complexes with Iron and Uranium.	Causes quench in liquid scintillation cocktails. ^{14}C might be lost as $^{14}CO_2$. Might cause corrosion of stainless steel planchets on gross analyses.
Addition of Sulfite	Forms a reducing environment to prevent the volatilization of iodine.	May produce undesirable oxidation states of iron or uranium.
Addition of Formaldehyde	Preserves organic samples. Prevents further biological activity.	May create disposal problems.
Cooling (Ice at approximately 0 °C)	Preserves organic samples (i.e., water, foods). Reduces dehydration and retains moisture. Reduces biological activity.	Ice melts, requiring replacement over time.
Freezing (Dry Ice at approximately -78 °C)	Preserves organic samples (i.e., water, plant, animal). Suspends biological activity.	Dry ice sublimates and requires replacement. May crack sample container if frozen too quickly.
Addition of Paper Pulp	Provides large surface area for adsorption of metals, thus minimizing adsorption on container walls.	Requires pH to be one or less. Requires filtration and wet ashing of paper pulp and combining liquids to make a new solution.

10.3.4 Liquid Samples: Special Cases

In some cases, liquid samples require special handling in order to preserve or retain a volatile or gaseous radionuclide. The following are examples of specific methods used to recover or preserve such samples of interest.

10.3.4.1 Radon-222 in Water

Waterborne radon is analyzed most commonly by liquid scintillation methods, although gamma-ray spectrometry and other methods have been employed or proposed. Liquid scintillation has the

obvious advantage of being designed for automated sample processing and is, therefore, less labor intensive or costly. A key to consistency in analytical results is the zero headspace sampling protocol such as the one described below.

Since radon is inert and nonpolar, it diffuses through plastic more rapidly than glass. The use of plastic scintillation vials, therefore, leads to significant loss of radon in water (Whittaker, 1989; Hess and Beasley, 1990). For this reason, it is recommended that the water sample is collected in a 23 mL glass scintillation vial, capped with a Teflon™ or foil-lined cap.

Samples are collected from a nonaerated faucet or spigot, which has been allowed to flow for sufficient time so that the sample is representative of the water in the distribution system or well. The time will vary depending on the source.

10.3.4.1 Milk

Milk commonly is viewed as the food product of greatest potential dose significance for airborne releases of radionuclides. Due to the animals' metabolic discrimination, however, only a few radionuclides have a significant dose impact via the milk pathway, notably ^{90}Sr, ^{131}I, and ^{137}Cs.

To prevent milk from souring or curdling, samples should be refrigerated. Preservation of milk may also be achieved through the addition of formaldehyde or methanol (DOE, 1987), methimazole (Harrington et al., 1980), or Thimerosal (EPA, 1994). Analytical procedures for select radionuclides in milk are well established and should be considered when deciding on a sample preservation method. Adding formaldehyde to milk samples may require them to be disposed of as hazardous or mixed wastes.

Due to the volatility and potential loss of ^{131}I (as I_2), a known amount of NaI dissolved in water may be added to the milk sample at time of collection if iodine analysis is required. The NaI not only serves as a carrier for the chemical separation of radioiodine, but also provides a quantitative tool for determining any loss prior to analysis (DOE, 1990).

10.3.5 Nonaqueous Liquids and Mixtures

Nonaqueous liquids and mixtures include a wide range of organic fluids or solvents, organic materials dissolved in water, oils, lubricants, etc. These liquids are not likely to represent contaminated environmental media or matrices, but most likely represent waste streams that must be sampled. Nonaqueous waste streams are generated as part of normal operations by nuclear utilities, medical facilities, academic and research facilities, state and federal agencies, radio-pharmaceutical manufacturers, DOE weapons complexes, mining and fuel fabrication facilities, etc. Examples of these nonaqueous liquids and mixtures include waste oils and other lubricants that are generated routinely from maintenance of equipment associated with nuclear power plant operations or the production of nuclear fuel and nuclear weapon components; and organic and

inorganic solvents, acids, and bases that are used in a variety of medical, research, and industrial applications.

In addition to the production of nonaqueous liquid wastes from routine operations by these facilities, large quantities of nonaqueous liquids containing radionuclide contaminants are also generated by routine facility decontamination efforts and final decontamination associated with facility decommissioning. For decontamination and decommissioning activities, a wide range of processes have been developed that employ halogenated organic compounds, such as Freon®, chloroform, or trichloroethane. Other aggressive chemical decontamination processes involve dissolution and removal of metal and oxide layers from surfaces using acid solutions (e.g., sulfuric acid, nitric acid, phosphoric acids, and oxalic acid). Chemical decontamination also may use chelating agents in concentrated processes (5 to 25 percent by weight chemical in solution) and dilute processes (one percent wt. or less chemicals in solution). Examples of chemical processes that can be used in both concentrated and dilute forms include the low oxidation-state transition-metal ion (LOMI) and LOMI-nitric permanganate, developed by Dow Chemical Company and AP/Citron. The reagents used in both the concentrated and dilute processes include chelating and complexing agents such as ethylene diamine tetraacetic acid (EDTA), diethylene triamine pentaacetic acid (DTPA), citric acid, oxalic acid, picolinic acid, and formic acid. Chelating agents and organic acids are used in decontamination formulas because they form strong complexes with actinides, lanthanides, heavy metals, and transition metals and assist in keeping these elements in solution.

Generally, these chemical decontamination solutions, once used, are treated with ion-exchange resins to extract the soluble activity. The ion-exchange decontamination solutions must be sampled, nevertheless, to assess the amount of residual radioactivity.

The radionuclides that may be encountered with nonaqueous liquids and mixtures depend on both the nature of the liquid and its usage. The following listing of radionuclides and liquids are based on published data collected by NRC (1992) and the State of Illinois (Klebe 1998; IDNS 1993-1997), but are not intended to represent a comprehensive list:

- Toluene/xylene/scintillation fluids used by research and clinical institutions: ^{3}H, ^{14}C, $^{32/33}P$, ^{35}S, ^{45}Ca, ^{63}Ni, ^{67}Ga, $^{125/131}I$, ^{99}Tc, ^{90}Sr, ^{111}In, $^{123/125}I$, ^{147}Pm, $^{201/202}Tl$, $^{226/228}Ra$, $^{228/230/232}Th$, $^{232/234/235/238}U$, $^{238/239/241/242}Pu$, ^{241}Am.

- Waste oils and lubricants from operation of motors, pumps, and other equipment: ^{3}H, ^{54}Mn, ^{65}Zn, ^{60}Co, $^{134/137}Cs$, $^{228/230/232}Th$.

- Halogenated organic and solvents from refrigeration, degreasing, and decontamination: ^{3}H, ^{14}C, $^{32/33}P$, ^{35}S, ^{54}Mn, $^{58/60}Co$, ^{63}Ni, ^{90}Sr, $^{125/129}I$, $^{134/137}Cs$, $^{226/228}Ra$, $^{228/230/232}Th$, $^{232/234/235/238}U$, $^{238/239/241}Pu$.

- Other organic solvents from laboratory and industrial operations and cleaning: ^{3}H, $^{32/33}P$, ^{35}S, ^{45}Ca, ^{125}I, U-natural.

- Inorganic and organic acids and bases from extraction processes and decontamination: ^{3}H, ^{14}C, $^{32/33}P$, ^{35}S, ^{54}Mn, ^{67}Ga, $^{125/131}I$, ^{60}Co, ^{137}Cs, and U-natural.

Due to the large number of potential nonaqueous liquids and the complex mixtures of radionuclide contaminants that may require radiochemical analysis, a comprehensive discussion of sample preparation and preservation is beyond the scope of this discussion. In most instances, however, these samples are not likely to require refrigeration or chemical preservatives that protect against sample degradation.

Some organic solvents and highly acidic or basic liquids may react with plastic containers, causing brittleness or breakage. In selecting sample containers for these nonaqueous samples, it is important to assess the manufacturers product specifications, which typically provide information regarding the container's resistance to chemical and physical agents. When nonaqueous samples are stored for long periods of time, containers should be checked routinely.

10.4 Solids

Solid samples consist of a wide variety of materials that include soil and sediment, plant and animal tissue, metal, concrete, asphalt, trash, etc. In general, most solid samples do not require preservation, but require specific processing in the field before transporting to the laboratory for analysis. For example, soil sample field processing may require sieving in order to establish sample homogeneity. These and other specific handling requirements are described below in the section on each type of solid sample.

The most critical aspect is the collection of a sufficient amount of a representative sample. One purpose of soil processing is to bring back only that sample needed for the laboratory. Unless instructed otherwise, samples received by the laboratory are typically analyzed exactly as they are received. This means that extraneous material should be removed at the time of sample collection, if indicated in the appropriate plan document.

In many instances, sample moisture content at the time of collection is an important factor. Thus, the weights of solid samples should be recorded at the time a sample is collected. This allows one to track changes in wet weight from field to laboratory. Dry and ash weights generally are determined at the laboratory.

Unlike liquid samples that may be introduced or removed from a container by simple pouring, solid samples may require a container that is designed for easy sample placement and removal. For this reason, large-mouth plastic containers with screw caps or individual boxes with sealable

plastic liners are commonly used. The containers also minimize the risk for breakage and sample cross-contamination.

10.4.1 Soils

ASTM D653 defines soil as: "Sediments or other unconsolidated accumulations of solid particles produced by the physical and chemical degradation of rocks, and that might or might not contain organic matter." ASTM C999 provides generic guidance for soil sample preparation for the determination of radionuclides. ASTM D4914 and D4943 provide additional information on soil and rock.

The distribution of radionuclides in soil should be assumed to be heterogeneous. The degree of heterogeneity is dictated by the radionuclide's mode of entry into the environment and soil, the chemical characteristics of the radionuclide contaminant, soil composition, meteorological and environmental conditions, and land use. For example, soil contamination from an airborne release of a radionuclide with strong affinity for clay or other mineral constituents of soil likely will exhibit a gradient with rapidly diminishing concentrations as a function of soil depth (the parameter associated with this affinity is K_D, which is the concentration of the solid phase divided by the concentration of the liquid phase). Moreover, contamination may be differentially distributed among soil particles of different sizes. In most cases, because the contaminant is adsorbed at the surface of soil particles and since the surface-to-volume ratio favors smaller particles, smaller soil particles will exhibit a higher specific activity when compared to larger particles. If land areas include areas of farming, tilling of soil will clearly impact the distribution of surface contamination.

10.4.1.1 Soil Sample Preparation

Extraneous material should be removed at the time of sample collection, if indicated in the appropriate plan document. The material may have to be saved and analyzed separately, depending on the project requirements and MQOs. If rocks, debris, and roots are removed from a soil sample after it arrives at the laboratory, there may be insufficient material to complete all the requested analyses (see Section 12.3.1.1 "Exclusion of Material"). A sufficient amount of sample should be collected to provide the net quantity necessary for the analysis. Subsequent drying at the laboratory may remove a large percentage of the sample weight that is available for analysis. Field-portable balances or scales may be used to weigh samples as they are collected, further ensuring sufficient sample weights are obtained. For certain types of samples, the project DQOs may require maintaining the configuration of the sample, such as core samples where concentration verses depth will be analyzed.

The project plan should address the impact of heterogeneity of radionuclide distribution in soil. Some factors to consider that may impact radionuclide distribution are: determining sampling depth, the need for removal of vegetative matter, rocks, and debris, and the homogenation of soil

particulates. For example, soil sampling of the top 5 cm is recommended for soils contaminated by recent airborne releases (ASTM C998); soil depth to 15 cm may be appropriate when exposure involves the need to monitor the root zone of food crops (MARSSIM, 2000; NRC, 1990). The need for sample field QC, such as splitting, should be evaluated. Some types of field QC can be used to evaluate the extent of radionuclide homogeneity. In general, no special preservation measures are required for soil samples; however, preliminary soil sample preparation involving drying, sieving, homogenizing, and splitting may be performed by a field laboratory prior to sample shipment to the analytical laboratory.

If volatile elements are suspected to be present with other nonvolatile contaminants, samples must be split before drying to avoid loss of the contaminant of interest. Dried samples are homogenized by mortar and pestle, jaw crusher, ball mill, parallel plate grinder, blender, or a combination of these techniques and sieved to obtain a uniform sample. Sieve sizes from 35 to 200 mesh generally are recommended for wet chemistry procedures. ASTM C999 correlates various mesh sizes with alternative designations, inclusive of physical dimensions expressed in inches or in the metric system. In addition, samples for chemical separations are usually ashed in a muffle furnace to remove any remaining organic materials that may interfere with the procedures.

10.4.1.2 Sample Ashing

Soil samples that require chemical separation for radionuclide analysis may also be ashed by the field laboratory. The use of the term "field laboratory" can cause confusion, since no single definition is possible. It is used here to define a laboratory that is close to the point of sample collection. It does not imply that there is a distinction in requirements or specifications that impact quality. For soil samples, ashing is performed in a muffle furnace to remove any organic materials that may interfere with radiochemical procedures.

10.4.2 Sediments

Sediments of lakes, reservoirs, cooling ponds, settling basins, and flowing bodies of surface water may become contaminated as a result of direct liquid discharges, wet surface deposition, or from runoffs associated with contaminated soils. Because of various chemically and physically binding interactions with radionuclides, sediments serve as integrating media that are important to environmental monitoring. An understanding of the behavior of radionuclides in the aquatic environment is critical to designing a sampling plan, because their behavior dictates their distribution and sampling locations.

In most cases, sediment is separated from water by simple decanting, but samples also may be obtained by filtering a slurry or through passive evaporation. As noted previously, care must be taken to avoid cross contamination from sampling by decontaminating or replacing tools and also from avoiding contact between successive samples. Suitable sample containers include glass or

plastic jars with screw caps. The presence of volatile or semi-volatile organic and micro-organisms may impact the radionuclide concentration, therefore, samples should be kept on ice while in the field and refrigerated while awaiting radioanalysis. Sediment cores may be sampled, frozen, and then sectioned.

10.4.3 Other Solids

10.4.3.1 Structural Materials

In some cases, a project plan requires sample analysis of structural materials such as concrete or steel. Concrete from floors, walls, sidewalks or road surfaces is typically collected by scabbling, coring, drilling, or chiseling. Depending on the radionuclides of interest and detection methods, these sample preparations may require crushing, pulverization, and sieving.

Metal associated with structures (e.g., I-beams, rebar) or machines may be contaminated on exterior or interior surfaces or through activation may become volumetrically contaminated. Surface contamination may be assessed by swipe samples that provide a measure of removable contamination (Section 10.6) or by scraping, sandblasting, or other abrasive techniques. Volumetric contamination is frequently assessed by nondestructive field measurements that rely on gamma-emitting activation products. However, drill shavings or pieces cut by means of a plasma arc torch may be collected for further analysis in a laboratory where they can be analyzed in a low-background environment. In general, these materials require no preservation but, based on activity/dose-rate levels and sample size and weight, may require proper shielding, engineered packaging, and shipping by a licensed carrier.

10.4.3.2 Biota: Samples of Plant and Animal Products

The release of radionuclides to the environment from normal facility operations or as the result of an accident requires the sampling of a wide variety of terrestrial and aquatic biota. For most biota, sample preservation usually is achieved by icing samples in the field and refrigeration until receipt by the analytical laboratory. The field sampling plan should describe the type of processing and preservation required.

Foods may be categorized according to the U.S. Department of Agriculture scheme as leafy vegetables, grains, tree-grown fruits, etc., and representative samples from each group may be selected for analysis.

MEAT, PRODUCE, AND DAIRY PRODUCTS

Samples of meat, poultry, eggs, fresh produce, and other food should be placed in sealed plastic bags and appropriately labeled and preserved by means of ice in the field and refrigeration during interim storage prior to delivery to the analytical laboratory. All food samples may be reduced to

edible portions (depending on study objective) for analysis in a manner similar to that for human consumption (i.e., remove cores, bones, seeds, other nonedible parts) and weighed as received from the field (i.e., wet weight) within 24 hours. Wet weights are desired, since consumption data are generally on this basis.

ANIMAL FEED AND VEGETATION

Animal feeds also provide important data for determining radionuclide concentrations in the food chain. Crops raised for animal feed and vegetation consumed by grazing farm animals may be sampled. Depending upon radionuclides under investigation and their associated MQOs, kilogram quantities of vegetative matter may be needed.

As in all terrestrial samples, naturally occurring ^{40}K and the uranium and thorium series radionuclides contribute to the radiation observed. Deposition of such cosmic-ray-produced nuclides as ^{7}Be and fallout from nuclear tests also may be present. Properly selected processed items from commercial sources may be helpful in providing natural and anthropogenic background data.

TERRESTRIAL WILDLIFE

Wild animals that are hunted and eaten may be of interest for potential dose estimates and therefore may require sampling. Examples of wildlife that have been used are deer, rabbits, and rodents that may feed or live in a contaminated site. An estimate of the radionuclide intake of the animal just before its death may be provided by analyzing the stomach content, especially the rumen in deer.

AQUATIC ENVIRONMENTAL SAMPLES

In addition to natural radionuclides and natural radionuclides enhanced by human activity, there are numerous man-made radionuclides that have the potential for contaminating surface and ground water. The most common of these are fission and activation products associated with reactor operation and fuel cycle facilities. Radioanalysis of aquatic samples may therefore include ^{54}Mn, ^{58}Co, ^{60}Co, ^{65}Zn, ^{95}Zr, ^{90}Sr, ^{134}Cs, ^{137}Cs, and transuranics, such as ^{239}Pu.

When surface and ground waters are contaminated, radionuclides may be transferred through a complex food web consisting of aquatic plants and animals. Aquatic plants and animals, as discussed here, are any species which derive all or substantial portions of their nourishment from the aquatic ecosystem, are part of the human food chain, and show significant accumulation of a radionuclide relative to its concentration in water. Although fish, aquatic mammals, and waterfowl provide a direct link to human exposure, lower members of the food chain also may be sampled.

FLORA

Aquatic biota such as algae, seaweed, and benthic organisms are indicators and concentrators of radionuclides—especially ^{59}Fe, ^{60}Co, ^{65}Zn, ^{90}Sr, ^{137}Cs, and the actinides—and can be vectors in the water-fish-human food chain. As such, they may be sampled upstream and downstream at locations similar to those described for sediment. Because of their high water content, several kilograms (wet weight) should be collected per sample. The wet weight of the sample should be recorded. Enough of the wet sample should be processed so that sufficient sample remains following the drying process. Both algae (obtained by filtering water or by scraping submerged substrates) and rooted aquatic plants should be sampled.

FISH AND SHELLFISH

Several kilograms of each fish sample are usually required; this may be one large fish, but preferably a composite of a number of small ones. Analysis of the edible portions of food fish as prepared for human consumption is of major interest. Fish may be de-boned, if specified in the sampling plan. The whole fish is analyzed if it is used for the preparation of a fish meal for consumption or if only trend indication is required. In a program where fish are the critical pathway, fish are analyzed by species; if less detail is required, several species with similar feeding habits (such as bottom feeders, insectivores, or predators) may be collected and the data grouped. Some species of commercial fish, though purchased locally, may have been caught elsewhere. Thus, the presence or absence of a radionuclide in a specific fish may not permit any definite conclusion concerning the presence of the radionuclide in water at that location.

Shellfish, such as clams, oysters, and crabs, are collected for the same reasons as fish, but have the advantage as indicators of being relatively stationary. Their restricted mobility contributes substantially to the interpretation and application of analytical results to environmental surveillance. Edible and inedible portions of these organisms can be prepared separately.

WATERFOWL

Waterfowl, such as ducks and geese, may also concentrate radionuclides from their food sources in the aquatic environment and serve as important food sources to humans. The migratory patterns and feeding habits of waterfowl vary widely. Some species are bottom feeders and, as such, tend to concentrate those radionuclides associated with sediments such as ^{60}Co, ^{65}Zn, and ^{137}Cs. Others feed predominantly on surface plants, insects, or fish.

An important consideration in obtaining a sample from waterfowl is that their exterior surfaces, especially feathers, may be contaminated. It is important to avoid contaminating the "flesh" sample during handling. As with other biota samples, analyses may be limited to the edible portions and should be reported on a wet weight basis.

10.5 Air Sampling

The measurement of airborne radionuclides as gases or particulates provides a means of evaluating internal exposure through the inhalation pathways. The types of airborne radioactivity that may require air sampling are normally categorized as: (1) airborne particulates; (2) noble gases; (3) volatilized halogens (principally radioiodines); and (4) tritiated water. Depending upon the source term and the objectives of the investigation, air sampling may be conducted outdoors as well as indoors on behalf of a variety of human receptors. For example, routine outdoor air samples may be taken for large population groups living within a specified radius of a nuclear facility. On the other end of the spectrum, air samples may be taken for a single person or small group of persons exposed occupationally to a highly localized source of airborne radioactivity.

The purpose of the samples being collected must, therefore, be well defined in terms of sampling location, field sampling equipment, and required sample volumes. Due to the wide range of conditions that may mandate air sampling, and the limited scope of this section, only generic topics of air sampling will be discussed.

10.5.1 Sampler Components and Operation

Common components of air sampling equipment include a sample collector (i.e., filter), a sample collector holder, an air mover, and a flow-rate measuring device.

The sample holder should provide adequate structural support while not damaging the filter, should prevent sampled air from bypassing the filter, should facilitate changing the filter, and should facilitate decontamination. A backup support that produces negligible pressure drop should be used behind the filter to prevent filter distortion or deterioration. If rubber gaskets are used to seal the filter to the backing plate, the gasket should be in contact with the filter along the entire circumference to ensure a good fit.

Air movers or vacuum systems should provide the required flow through the filter and minimize air flow reduction due to filter loading. Consideration should be given to the use of air movers that compensate for pressure drop. Other factors to consider should include size, power consumption, noise, durability, and maintenance requirements.

Each air sampler should be equipped with a calibrated air-flow measuring device with specified accuracy. To calculate the concentrations of any radionuclide in air collected, it is necessary to determine the total volume of air sampled and the associated uncertainties. The planning documents should state who is responsible for making volume corrections. Also, the information needed for half-life corrections for short-lived radionuclides needs to be recorded. If the mean flow during a collection period can be determined, the total volume of air sampled can be readily calculated.

Accurate flow measurements and the total integrated sample volume of air can be obtained using a mass flow meter and a totalizer. This direct technique of air flow measurement becomes impractical at remote field locations, due to cost and exposure of the flow meter to harsh environments. Other procedures for the measurement of air flow in sampling systems are reviewed by Lippmann (1989a). The sample parameters (flow rate, volume, associated uncertainties, etc.) should be recorded by the sample collector.

The collection medium or filter used depends on the physical and chemical properties of the materials to be collected and counted. A variety of particulate filters (cellulose, cellulose-asbestos, glass fiber, membrane, polypropylene, etc.) is available. The type of filter is selected according to needs, such as high collection efficiency, particle-size selectivity, retention of alpha emitters on the filter surface, and the compatibility with radiochemical analysis. The criteria for filter selection are good collection efficiency for submicron particles at the range of face velocities used, high particle and mass loading capacity, low-flow resistance, low cost, high mechanical strength, low-background activity, compressibility, low-ash content, solubility in organic solvents, nonhygroscopicity, temperature stability, and availability in a variety of sizes and in large quantities. The manufacturer's specifications and literature should provide a source for filter collection efficiency. In the selection of a filter material, a compromise must be made among the above-cited criteria that best satisfies the sampling requirements. An excellent review of air filter material used to monitor radioactivity was published by Lockhart and Anderson (1964). Lippmann (1989b) also provides information on the selection of filter materials for sampling aerosols by filtration. See ANSI HPS N13.1, Annex D and Table D.1, for criteria for the selection of filters for sampling airborne radioactive particles.

In order to select a filter medium with adequate collection efficiency, it may be necessary to first determine the distribution of size of airborne particulates. Several methods, including impactors (e.g., multistage cascade impactor) and electrostatic precipitators, can be used to classify particle size. Waite and Nees (1973) and Kotrappa et al. (1974) discuss techniques for particle sizing based on the flow discharge perturbation method and the HASL cyclone, respectively. These techniques are not recommended for routine environmental surveillance of airborne particulates, although their use for special studies or for the evaluation of effluent releases should not be overlooked. Specific data on various filter materials, especially retention efficiencies, have been reported by several authors (Lockhart and Anderson, 1964; Denham, 1972; Stafford, 1973; ASTM STP555) and additional information is available from manufacturers.

10.5.2 Filter Selection Based on Destructive Versus Nondestructive Analysis

Pure cellulose papers are useful for samples to be dissolved and analyzed radiochemically, but the analytical filter papers used to filter solutions are inefficient collectors for aerosols and clog easily. Cellulose-asbestos filter papers combine fairly high efficiency, high flow rates, high mechanical strength, and low pressure drops when loaded. They are very useful for collecting large samples but present difficulties in dissolution, and their manufacture is diminishing because

of the asbestos. Fiberglass filters can function efficiently at high flow rates, but require fluoride treatment for dissolution and generally contain sufficient radioactive nuclides to complicate low-activity analysis. Polystyrene filters are efficient and capable of sustaining high air flow rates without clogging. They are readily destroyed for analysis by ignition (300 °C) or by wet washing with oxidizing agents, and also are soluble in many organic liquids. They have the disadvantage of low mechanical and tensile strength, and they must be handled carefully. Membrane filters are excellent for surface collection efficiency and can be used for direct alpha spectrometry on the filter. However, they are fragile and suffer from environmental dust loading. An alternative choice for radionuclides in the environment is the polypropylene fiber filter. Teflon™ fiber filters can be efficient, but they should be used with care because of their high ashing temperatures and difficulties with digestion.

10.5.3 Sample Preservation and Storage

Since particulate air samples are generally dry samples that are chemically and physically stable, they require no preservation. However, care must be exercised to avoid loss of sample from the filter medium and the cross contamination among individual samples. Two common methods are to fold filters symmetrically so that the two halves of the collection surface are in contact, or to insert the filter into glassine envelopes. Filters should be stored in individual envelopes that have been properly labeled. Filters may also be stored in special holders that attach on the filter's edge outside of the collection surface.

Since background levels of ^{222}Rn and ^{220}Rn progeny interfere with evaluating alpha air samples, a holdup time of several hours to several days may be required before samples are counted. Corrections or determinations can also be made for the contribution of radon or thoron progeny present on a filter (Setter and Coats, 1961).

10.5.4 Special Cases: Collection of Gaseous and Volatile Air Contaminants

Prominent radionuclides that may exist in gaseous states include noble gases (e.g., $^{131/133}$Xe, ^{85}Kr), ^{14}C as carbon dioxide or methane, ^{3}H as water vapor, gaseous hydrogen, or combined in volatile organic compounds and volatilized radioiodines.

10.5.4.1 Radioiodines

The monitoring of airborne iodine, such as ^{129}I and ^{131}I, may be complicated by the probable existence of several species, including particulate iodine or iodine bound to foreign particles, gaseous elemental iodine, and gaseous non-elemental compounds of iodine. A well-designed sampling program should be capable of distinguishing all possible iodine forms. While it may not always be necessary to differentiate between the various species, care should be taken so that no bias can result by missing one or more of the possible species. See ANSI HPS N13.1 (Annex C.3) for information on collection media for radioiodine.

In addition to the problems noted above, charcoal cartridges (canisters) for the collection of radioiodine in air are subject to channeling. Several should be mounted in series to prevent loss of iodine. Too high a sampling rate reduces both the collection efficiency and retention time of charcoal filters, especially for the non-elemental forms of iodine (Keller et al., 1973; Bellamy, 1974). The retention of iodine in charcoal is dependent not only on charcoal volume, but also the length of the charcoal bed. Typical air flow rates for particulate sampling of 30 to 90 L/min (1 to 3 ft^3/min) are normally acceptable for environmental concentrations of radioiodine. The method proposed by the Intersociety Committee (APHA, 1972) for ^{131}I concentrations in the atmosphere involves collecting iodine in its solid and gaseous states with an "absolute" particulate filter in series with an activated charcoal cartridge followed by gamma spectrometric analysis of the filter and cartridge. The Intersociety-recommended charcoal cartridges are ⅝ inch (16 mm) diameter by 1½ inch (38 mm) deep containing 3 g of 12-to-30-mesh KI-activated charcoal. The minimum detectable level using the Intersociety method is 3.7×10^{-3} Bq/m^3 (0.1 pCi/m^3). Larger cartridges will improve retention, permitting longer sampling periods. A more sensitive system has been described by Baratta et al. (1968), in which concentrations as low as 0.037 Bq/m^3 (0.01 pCi/mL) of air are attainable.

For the short-lived radioiodines (mass numbers 132, 133, 135), environmental sampling is complicated by the need to obtain a sufficient volume for analysis, while at the same time, retrieving the sample soon enough to minimize decay (with half-lives ranging from two hours to 21 hours). Short-period (grab) sampling with charcoal cartridges is possible, with direct counting of the charcoal as soon as possible for gamma emissions.

Because of the extremely long half-life and normally low environmental concentrations, ^{129}I determinations must usually be performed by neutron activation or mass spectrometry analysis after chemical isolation of the iodine. For concentrations of about 0.11 Bq/L (3×10^{-10} μCi/mL), liquid scintillation counting can be used after solvent extraction (Gabay et al., 1974).

10.5.4.2 Gases

Sampling for radioactive gases is either done by a grab sample that employs an evacuated chamber or by airflow through a medium, such as charcoal, water, or a variety of chemical absorbers. For example, radioactive CO_2 is most commonly extracted by passing a known volume of air through columns filled with 3 M NaOH solution. After the NaOH is neutralized with sulfuric acid, the CO_2 is precipitated in the form of $BaCO_3$, which then can be analyzed in a liquid scintillation counter (NCRP, 1985). An alternative method for collecting noble gases by compression into high-pressure canisters is described in Section 15.3.5.1, "Radioactive Gases."

Because noble gases have no metabolic significance, and concern is principally limited to external exposure, surveillance for noble gases is commonly performed by ambient dose rate measurements. However, the noble gases xenon and krypton may be extracted from air by adsorption on activated charcoal (Scarpitta and Harley, 1990). However, depending upon the

analytical method and instrumentation employed, significant interference may result from the presence of naturally occurring radioactive gases of ^{222}Rn and ^{220}Rn.

10.5.4.3 Tritium Air Sampling

In air, tritium occurs primarily in two forms: as water vapor (HOT) and as hydrogen gas (HT). However, if tritiated hydrogen (HT) is a suspected component of an air sample (e.g., from a vent or stack), the sampling must take place in the emission point of the gas. This is because the high escape velocity of hydrogen gas causes rapid, isotropic dispersion immediately beyond the discharge point. Tritiated organic compounds in the vapor phase or attached to particulate matter occur only occasionally. To measure tritium as HT or in tritiated organic, the gas phase can be oxidized, converting the tritium to HOT before desiccation and counting. For dosimetric purposes, the fraction present as HT can usually be neglected, since the relative dose for a given activity concentration of HOT is 400 times that for HT (NCRP, 1978). However, if HT analysis is required, it can be removed from the atmosphere by oxidation to water (HOT) using CuO/MnO_2 at 600 °C (Pelto et al., 1975), or with air passed over platinum alumina catalyst (Bixel and Kershner 1974). These methods also oxidize volatile tritiated organic compounds to yield tritiated water (ANSI HPS N13.1, Annex H).

A basic system for sampling HOT consists of a pump, a sample collector, and a flow-measuring or flow-recording device. Air is drawn through the collector for a measured time period at a monitored flow rate to determine the total volume of air sampled. The total amount of HOT recovered from the collector is divided by the total volume of air sampled to determine the average HOT-in-air concentration of the air sampled. In some sampler types, the specific activity of the water collected is measured and the air concentration is determined from the known or measured humidity. Some common collectors are cold traps, tritium-free water, and solid desiccants, such as silica gel, DRIERITE™, or molecular sieve.

Cold traps are usually made of glass and consist of cooled collection traps through which sample air flows. The trap is cooled well below the freezing point of water, usually with liquid nitrogen. The water vapor collected is then prepared for analysis, usually by liquid scintillation counting. Phillips and Easterly (1982) have shown that more than 95 percent HOT collection efficiency can be obtained using a single cold trap. Often a pair of cold traps is used in series, resulting in a collection efficiency in excess of 99 percent.

Gas-washing bottles (i.e., “bubblers”) filled with an appropriate collecting liquid (usually tritium-free water) are used quite extensively for collecting HOT from air. HOT in the sample gas stream “dissolves” in the collecting liquid. For the effective collection rate to remain the same as the sample flow rate, the specific activity of the bubbler water must be negligible with respect to the specific activity of the water vapor. Thus, the volume of air that can be sampled is ultimately limited by the volume of water in the bubbler. However, except when sampling under conditions of very high humidity, sample loss (dryout) from the bubbler usually limits collection time rather

than the attainment of specific-activity equilibrium. Osborne (1973) carried out a thorough theoretical and experimental evaluation of the HOT collection efficiency of water bubblers over a wide range of conditions.

The use of silica gel as a desiccant to remove moisture from air is a common technique for extracting HOT. The advantage of using silica gel is that lower HOT-in-air concentrations can be measured, since the sample to be analyzed is not significantly diluted by an initial water volume, which occurs when a liquid-sampling sink is used. Correcting for dilution is discussed in Rosson et al. (2000).

10.5.4.4 Radon Sampling in Air

There are three isotopes of radon in nature: ^{222}Rn is a member of the ^{238}U decay chain; ^{220}Rn is a member of the ^{232}Th decay chain; and ^{219}Rn is a member of the ^{235}U decay chain. Because of the small relative abundance of the parent nuclides and the short half-lives of ^{220}Rn (55 seconds) and ^{219}Rn (4 seconds), the term "radon" generally refers to the isotope ^{222}Rn. Owing to its ubiquitous presence in soils, uranium mill tailings, underground mines, etc., and the health risks to large populations and occupational groups, radon is perhaps the most studied radionuclide.

Consequently, many reports and articles have been published in the scientific literature dealing with the detection methods and health risks from radon exposures. Many of them appear in publications issued by the EPA, DOE, NCRP, NAS, and in radiation-related journals, such as *Health Physics* and *Radiation Research*. Given the voluminous amount of existing information, only a brief overview of the sampling issues that impact laboratory measurements can be presented here.

Quantitative measurements of radon gas and its short-lived decay products can be obtained by several techniques that are broadly categorized as grab sampling, continuous radon monitoring, and integrative sampling. Each method imposes unique requirements that should be followed carefully. Continuous monitors are not discussed further, since they are less likely to be used by laboratory analysts. Guidance for radon sample collection was published by EPA's Radon Proficiency Program, which was discontinued in October 1998 (EPA 1992; 1993). Additional sampling methods and materials are also presented in EPA (1994) and Cohen (1989).

In general, EPA's protocols specify that radon sampling and measurements be made under standardized conditions when radon and its progeny are likely to be at their highest concentrations and maximum equilibrium. For indoor radon measurement, this implies minimum building ventilation through restrictions on doors, windows, HVAC systems, etc. Also sampling should not take place during radical changes in weather conditions. Both high winds and rapid changes in barometric pressure can dramatically alter a building's natural ventilation rate. Although recommended measurements are likely to generate higher than actual average concentrations, the benefit of a standardized sampling condition is that it is reproducible, least variable, and

moderately conservative.

The choice among sampling methods depends on whether the measurement is intended as a short-term, quick-screening measurement or as a long-term measurement that determines average exposure or integration. In practice, the choice of a measurement system often is dictated by availability. If alternative systems are available, the cost or duration of the measurement may become the deciding factor. Each system has its own advantages and disadvantages, and the investigator must exercise some judgment in selecting the system best suited to the objectives of the investigation. Brief descriptions of several basic techniques used to sample air for radon and its progeny are provided below.

GRAB SAMPLING

The term "grab sampling" refers to very short-term sampling. This method consists of evaluating a small volume of air for either radon or radon decay product concentration. In the radon grab sampling method, a sample of air is drawn into and subsequently sealed in a flask or cell that has a zinc sulfide phosphor coating on its interior surfaces. One surface of the cell is fitted with a window that is put in contact with a photomultiplier tube to count light pulses (scintillations) caused by alpha disintegrations from the sample interacting with the zinc sulfide coating. The general terms "flask" or "cell" are used in this discussion. Sometimes they are referred to as "Lucas cells" (Lucas, 1982). The Lucas cell—or alpha scintillation counter—has specific attributes, and not all radon cells are Lucas cells.

Several methods for performing such measurements have been developed. However, two procedures that have been most widely used with good results are the Kusnetz procedure and the modified Tsivogiou procedure. In brief, the Kusnetz procedure (Kusnetz, 1956; ANSI N13.8) may be used to obtain results in working levels when the concentration of individual decay products is not important. Decay products in up to 100 liters of air are collected on a filter in a five-minute sampling period. The total alpha activity on the filter is counted any time between 40 and 90 minutes after sampling is completed. Counting can be done using a scintillation-type counter to obtain gross alpha counts for a selected counting time. Counts from the filter are converted to disintegrations using the appropriate counter efficiency. The disintegrations from the decay products may be converted into working levels using the appropriate "Kusnetz factor" for the counting time used.

The Tsivogiou procedure may be used to determine both working level and the concentration of the individual radon decay products. Sampling is the same as in the Kusnetz procedure. However, the filter is counted three separate times following collection. The filter is counted between 2 and 5 minutes, 6 and 20 minutes, and 21 and 30 minutes after sampling is complete. Count results are interpreted by a series of equations that calculate concentrations of the three radon decay products and working levels.

INTEGRATING SAMPLING DEVICES

By far, the most common technique for measuring radon is by means of integrating devices. Integrating devices, like charcoal canister and the Electret-Passive Environmental Radon Monitor (E-PERM®), are commonly employed as short-term integrating devices (two to seven days), while alpha-track detectors are commonly used to provide measurements of average radon levels over periods of weeks to months. Only charcoal canisters are discussed below, since they are more likely to be used by laboratory analysts than electrets and alpha-track detectors.

CHARCOAL CANISTERS

Charcoal canisters are passive devices requiring no power to function. The passive nature of the activated charcoal allows continual adsorption and desorption of radon. During the measurement period, the adsorbed radon undergoes radioactive decay. Therefore, the technique does not uniformly integrate radon concentrations during the exposure period. As with all devices that store radon, the average concentration calculated using the mid-exposure time is subject to error if the ambient radon concentration adsorbed during the first half of the sampling period is substantially higher or lower than the average over the period. The ability of charcoal canisters to concentrate noble gases or other materials may be affected by the presence of moisture, temperature, or other gaseous or particulate materials that may foul the adsorption surface of the charcoal.

10.6 Wipe Sampling for Assessing Surface Contamination

Surface contamination falls into two categories: fixed and loose. The wipe test (also referred to as "swipes" or "smears") is the universally accepted technique for detecting removable radioactive contamination on surfaces (Section 12.5, "Wipe Samples"). It is often a stipulation of radioactive materials licenses and is widely used by laboratory personnel to monitor their work areas, especially for low-energy radionuclides that are otherwise difficult to detect with hand-held survey instruments." Frame and Abelquist (1999) provide a comprehensive history of using smears for assessing removable contamination.

The purpose of the wipe test, organizational requirements or regulations, the nature of the contamination, the surface characteristics, and the radionuclide all influence the conditions for the actual wipe-test process. The wipe-test process should be standardized to ensure that the sampling process is consistent. Since surfaces and wipe materials vary considerably, wipe-test results provide qualitative indication of removable contamination. Fixed contamination will, by definition, not be removed. Therefore, direct measurements may be necessary to determine the extent on contamination.

The U.S. Nuclear Regulatory Commission (NRC, 1981) suggests that 100 cm^2 areas be wiped

and lists acceptable levels for surface contamination. However, NRC neither recommends the collection device nor the manner in which to conduct such surveys, relying instead on suggestions by the National Committee on Radiation Protection (1964) and the National Council on Radiation Protection and Measurements (1978).

To maintain constant geometry in an automatic proportional counter, it is important that the wipe remain flat during counting. Additionally, material that will curl can jam the automatic counter and cause cross contamination or even destroy the instrument window. When it is necessary to do destructive analysis on the wipe, it is critical that the wipe can easily be destroyed during the sample preparation step, and that the residue not cause interference problems.

When wipes are put directly into liquid scintillation cocktail, it is important that the wipe not add color or react with the cocktail. For maximum counting efficiency, as well as reproducibility, the wipe either should dissolve in the cocktail or become transparent to the counting system.

10.6.1 Sample Collection Methods

10.6.1.1 Dry Wipes

Dry wipes (smears) for removable surface activity usually are obtained by wiping an area of 100 cm^2 using a dry filter paper of medium hardness while applying moderate pressure. A 47 mm diameter filter typically is used. This filter can be placed into a proportional counter for direct counting. Smaller filters may be advantageous when the wipe is to be counted using liquid scintillation counter for low energy beta-emitting radionuclides, such as tritium, ^{14}C, and ^{63}Ni. The choice of wipe-test media and cocktail is critical when counting low-energy beta-emitting radionuclides in liquid scintillation counters, because the liquid scintillation counting process depends on the detection of light produced by the interaction of the radiation with the cocktail. The filter may absorb energy from the radiation (see "Quench" under Section 15.5.3.3). A filter that is in the cocktail can prevent light from being seen by both detectors at the same time. If light is produced and seen by only one of the two detectors typical in liquid scintillation counting systems, then the count will be rejected as noise. A filter/cocktail combination that produces a sample that is transparent to the counting system is the best combination for liquid scintillation counting. Background produced by the filter may also be a consideration.

For surveys of small penetrations, such as cracks or anchor-bolt holes, cotton swabs are used to wipe the area of concern. The choice of material for wipe-testing for special applications is critical (Hogue, 2002), and the material selected can significantly affect the efficiency of the removal of surface radioactivity. Usually, switching wipe test material should be avoided during a project, when possible. Samples (dry wipes or swabs) are placed into envelopes or other individual containers to prevent cross-contamination while awaiting analysis. Dry wipes for alpha and medium- or high-energy beta activity can be evaluated in the field by counting them on an integrating scaler unit with appropriate detectors; the same detectors utilized for direct

measurements may be used for this purpose. However, the more common practice is to return the dry wipes to the laboratory, where analysis can be conducted using more sensitive techniques. The most common method for analyzing wipe samples is to use a proportional counter. For very low-energy beta emissions, wipe samples are commonly analyzed by liquid scintillation counting.

Additional information on wipe-test counting can be found in ISO (7503-1; 7503-2; 7503-3), which apply to surfaces of equipment and facilities, containers of radioactive materials, and sealed sources. Abelquist (1998) discusses using smears to assess the quantity of removable contamination as it applies to radiological surveys in support of decommissioning, compliance with DOT shipping criteria, and operational radiological protection programs.

10.6.1.2 Wet Wipes

Although dry wipes are more convenient to handle, and there are fewer chances of cross contamination, a general limitation of dry wipes is their low recovery of surface contamination. The low recovery using dry wipes is due to the higher affinity for the surface by the contaminant than for the filter paper. Several studies have shown that for maximum sensitivity, a wipe material moistened with a suitable solvent may be indicated. For example, Ho and Shearer (1992) found that alcohol-saturated swabs were 100 times more efficient at removing radioactivity than dry swabs.

In another study, Kline et al. (1992) assessed the collection efficiency of wipes from various surfaces that included vinyl floor tile, plate glass, and lead foil. Two different collection devices, cotton swabs and 2.5 cm diameter glass fiber filter disks, were evaluated under various collection conditions. Dry wipes were compared to collections made with the devices dampened with different amounts of either distilled H_2O, 70 percent ethanol, or a working-strength solution of a multipurpose laboratory detergent known to be effective for removing contaminants from laboratory glassware (Manske et al., 1990).

The entire area of each square was manually wiped in a circular, inwardly-moving motion with consistent force. The collection capacity of each device was estimated by wiping progressively larger areas (multiple grids) and comparing the measured amounts of radioactivity with the amounts placed on the grids.

Collection efficiency varied with both the wipe method and the surface wipe. Contamination was removed most readily from unwaxed floor tile and glass; lead foil released only about one-half the radioactivity. Stainless steel, another common laboratory surface, has contamination retention properties similar to those of glass.

In most cases, collection was enhanced by at least a factor of two after dampening either the swabs or filter disks with water. Dampening with ethanol or the detergent produced removals that

were statistically indistinguishable from samples dampened with an equal amount of water.

The filter disks had a higher collection capacity for removable contaminants than cotton swabs, nearly doubling the radioactivity removed for each doubling of surface area wiped. Variability within all methods was high, with coefficients of variation ranging from 2 to 30 percent.

For the moistened wipes, wipe efficiency depended on three factors, including the polarity of the solvent, the polarity of the contaminant being measured, and the affinity of the compound for the contaminated surface. For a solvent to readily dissolve a compound (i.e., remove it from the surface), the solvent and the compound must have similar polarities. Nonpolar solvents include ethyl acetate and petroleum ether; for polar solvents, water or methanol may be used (Campbell et al., 1993). There are other factors that influence the affinity of a compound for a surface, including porosity of the surface and available binding sites on the surface. One important factor that influences binding capacity is the type of treatment that a surface has received. When working with a surface treated with a nonpolar wax, such as that used on floor tile, a nonpolar compound will be adsorbed to the surface, which further limits recovery. Recovery from absorbent surfaces, such as laboratory bench paper or untreated wood, also may be poor due to the porous nature of the surface.

10.6.2 Sample Handling

Filter paper or other materials used for wipe tests in the field should be placed in separate containers that prevent cross contamination during transport and allow for labeling of each sample. Plastic bags, paper or glassine envelopes, and disposable plastic petri dishes are typically used to store and transport wipe samples. Field workers can use plastic or rubber gloves and forceps when applying the wipe material to a surface and during handling as each wipe is placed into a container. Protection of the sample wipe surface is the main concern when a wipe must be placed in a container for transport. If a scintillation vial or planchet will be used in the laboratory, then a field worker may put wipes directly into them. Planchets containing loose or self-sticking wipes can also be put into self-sealing plastic bags to separate and protect the integrity of the sample's surface. Excessive dust and dirt can cause self adsorption or quenching, and therefore should be minimized.

10.6.3 Analytical Considerations for Wipe Material Selection

Some analytical considerations for selecting wipe materials are included here, because field sample collection and subsequent sample counting usually occur without such intervening steps as sample preparation, sample dissolution, or separation. It is critical, therefore, to ensure that the wipe material used for collection and the actual counting process are compatible. The following paragraphs offer some general guidance for proportional and liquid scintillation counting. The final paragraph discusses some key issues that impact dissolution of wipes.

The wipe should remain flat during counting in order to maintain optimum counting geometry in an automatic proportional counter. Wipe material that can curl may jam an automatic counter and destroy the detector window of the counter, become a source of cross-contamination of samples, or contaminate the counting system. Most proportional counting systems use two-inch (5 cm) planchets, and the wipe should fit into the planchet. If not, a subsample will need to be taken, and subsampling adds additional uncertainty due to sample homogeneity considerations.

When wipes are put directly into a liquid scintillation cocktail, the wipe should not add color or react with the cocktail. For maximum counting efficiency and reproducibility, the wipe either should dissolve or become transparent to the counting system. When wipes that have an adhesive backing are put directly in a liquid scintillation cocktail, the adhesive may not dissolve completely. Compatibility should be checked before use to prevent problems during actual sample analysis. Special cocktails are available to dissolve filters, but they may cause a waste-disposal problem. Since the possible combination of cocktails and filters is large, only general guidance is provided here. Consult the manufacturer's specifications for specific guidance.

When it is necessary to do destructive analysis on a wipe, select a wipe that can be destroyed easily or dissolved during the sample preparation steps, and the residue will not cause interference problems in the subsequent counting. Some wipes have adhesive backing; the wipe materials may dissolve easily but the adhesive backing may not. Additional steps would then be necessary to destroy the adhesive backing. Dissolving glass-fiber wipes may require the use of hydrofluoric acid. These extra processes can add time or cost to the analysis. See Section 10.5.2 ("Filter Selection Based on Destructive Versus Nondestructive Analysis"), Section 12.5 ("Wipe Samples") and Chapter 13 (*Sample Dissolution*) for additional information.

10.7 References

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