7 SAMPLING AND PREPARATION FOR LABORATORY MEASUREMENTS

7.1 Introduction

There are three methods for collecting radiation data while performing a survey. A direct measurement is obtained by placing the detector near or against the surface or in the media being surveyed and reading the radioactivity level directly. Scanning is an evaluation technique performed by moving a portable radiation detection instrument at a constant speed and distance above the surface to semi-quantitatively detect elevated areas of radiation. These measurement techniques are discussed in Chapter 6. Sampling is the process of collecting a portion of an environmental medium as representative of the locally remaining medium. The collected portion of the medium is then analyzed to determine the radionuclide concentration. This chapter discusses issues involved in collecting and preparing samples in the field for analysis, and in evaluating the results of these analyses. In addition, a general discussion on laboratory sample preparation and analysis is provided to assist in communications with the laboratory during survey planning.

Samples should be collected and analyzed by qualified individuals using the appropriate equipment and procedures. This manual assumes that the samples taken during the survey will be submitted to a qualified laboratory for analysis. The laboratory should have written procedures that document its analytical capabilities for the radionuclides of interest and a Quality Assurance/Quality Control (QA/QC) program that documents the compliance of the analytical process with established criteria. The method used to assay for the radionuclides of concern should be recognized as a factor affecting analysis time.

Commonly used radiation detection and measuring equipment for radiological survey field applications is described in Chapter 6 and Appendix H. Many of these equipment types are also used for laboratory analyses, usually under more controlled conditions that provide for lower detection limits and greater delineation between radionuclides. Laboratory methods often involve combinations of both chemical and instrument techniques to quantify the low levels expected in the samples. This chapter provides guidance to assist the MARSSIM user in selecting appropriate procedures for collecting and handling samples for laboratory analysis. More detailed information is available in documents listed in the reference section of this manual.

7.2 Data Quality Objectives

The survey design is developed and documented using the Data Quality Objectives (DQO) Process (see Appendix D). The third step of the DQO Process involves identifying the data needs for a survey. One decision that can be made at this step is the selection of direct

measurements for performing a survey or deciding that sampling methods followed by laboratory analysis are necessary.

7.2.1 Identifying Data Needs

The decision maker and the survey planning team need to identify the data needs for the survey being performed, including the:

- type of samples to be collected or measurements to be performed (Chapter 5)
- radionuclide(s) of interest (Section 4.3)
- number of samples to be collected (Section 5.5.2)
- type and frequency of field QC samples to be collected (Section 4.9)
- amount of material to be collected for each sample (Section 4.7.3 and Section 7.5)
- sampling locations and frequencies (Section 5.5.2)
- standard operating procedures (SOPs) to be followed or developed (Chapter 7)
- analytical bias and precision (*e.g.*, quantitative or qualitative) (Appendix N)
- target detection limits for each radionuclide of interest (Section 6.4 and Table 7.2)
- cost of the methods being evaluated (cost per analysis as well as total cost) (Appendix H)
- necessary turnaround time
- sample preservation and shipping requirements (Section 7.6 and Section 7.9)
- specific background for the radionuclide(s) of interest (Section 4.5)
- derived concentration guideline level (DCGL) for each radionuclide of interest (Section 4.3)
- measurement documentation requirements (Section 9.4.2.2)
- sample tracking requirements (Section 7.8)

Some of this information will be supplied by subsequent steps in the DQO process, and several iterations of the process may be needed to identify all of the data needs. Consulting with a radiochemist or health physicist may be necessary to properly evaluate the information before deciding between direct measurements or sampling methods to perform the survey. Surveys may require data from all three collection methods (*i.e.*, sample analysis, direct measurements, and scans) in order to demonstrate compliance with the regulation.

7.2.2 Data Quality Indicators

The data quality indicators identified as DQOs in Section 2.3.1 and described in Appendix N, Section N.6, should be considered when selecting a measurement method (*i.e.*, scanning, direct measurement, sampling) or an analytical technique (*e.g.*, radionuclide-specific analytical procedure). In some instances, the data quality indicator requirements will help in the selection of an analytical technique. In other cases, the analytical requirements will assist in the selection of appropriate levels for the data quality indicators.

7.2.2.1 Precision

Precision is a measure of agreement among replicate measurements of the same property under prescribed similar conditions (ASQC 1995). Precision is determined quantitatively based on the results of replicate measurements (equations are provided in EPA 1990). The number of replicate analyses needed to determine a specified level of precision for a project is discussed in Section 4.9. There are several types of replicate analyses available to determine the level of precision, and these replicates are typically distinguished by the point in the sample collection and analysis process where the sample is divided. Determining precision by replicating measurements with results at or near the detection limit of the measurement system is not recommended because the measurement uncertainty is usually greater than the desired level of precision.

- Collocated Samples. Collocated samples are samples collected adjacent to the routine field sample to determine local variability of the radionuclide concentration. Typically, collocated samples are collected about one-half to three feet away from the selected sample location. Analytical results from collocated samples can be used to assess site variation, but only in the immediate sampling area. Collocated samples should not be used to assess variability across a site and are not recommended for assessing error (EPA 1991g). Collocated samples can be non-blind, single-blind, or double-blind.

- Field Replicates. Field replicates are samples obtained from one location, homogenized, divided into separate containers and treated as separate samples throughout the remaining sample handling and analytical processes. These samples are used to assess error associated with sample heterogeneity, sample methodology and analytical procedures. Field replicates are used when determining total error for critical samples with contamination concentrations near the action level. For statistical analysis to be valid in such a case, a minimum of eight replicate samples would be required (EPA 1991g). Field replicates (or field split samples) can be non-blind, single-blind, or double-blind and are recommended for determining the level of precision for a radiation survey or site investigation.

- Analytical Laboratory Replicate. An analytical laboratory replicate is a subsample of a routine sample that is homogenized, divided into separate containers, and analyzed using the same analytical method. It is used to determine method precision, but because it is a non-blind sample, or known to the analyst, it can only be used by the analyst as an internal control tool and not as an unbiased estimate of analytical precision (EPA 1990).

- Laboratory Instrument Replicate. A laboratory instrument replicate is the repeated measurement of a sample that has been prepared for counting (*i.e.*, laboratory sample preparation and radiochemical procedures have been completed). It is used to determine

precision for the instrument (repeated measurements using same instrument) and the instrument calibration (repeated measurements using different instruments, such as two different germanium detectors with multichannel analyzers). A laboratory instrument replicate is generally performed as part of the laboratory QC program and is a non-blind sample. It is typically used as an internal control tool and not as an unbiased estimate of analytical precision.

7.2.2.2 Bias

Bias is the systematic or persistent distortion of a measurement process that causes error in one direction (ASQC 1995). Bias is determined quantitatively based on the analysis of samples with a known concentration. There are several types of samples with known concentrations. QC samples used to determine bias should be included as early in the analytical process as possible.

- Reference Material. A material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (ISO 1993). A certified reference material is reference material for which each certified property value is accompanied by an uncertainty at a stated level of confidence. Radioactive reference materials may be available for certain radionuclides in soil (*e.g.*, uranium in soil), but reference building materials may not be available. Because reference materials are prepared and homogenized as part of the certification process, they are rarely available as double-blind samples. When appropriate reference materials are available (*i.e.*, proper matrix, proper radionuclide, proper concentration range), they are recommended for use in determining the overall bias for a measurement system.

- Performance Evaluation (PE) Samples. PE sample are samples that evaluate the overall bias of the analytical laboratory and detect any error in the analytical method used. These samples are usually prepared by a third party, using a quantity of analyte(s) which is known to the preparer but unknown to the laboratory, and always undergo certification analysis. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of interest. Laboratory procedural error is evaluated by the percentage of analyte identified in the PE sample (EPA 1991g). PE samples are recommended for use in determining overall bias for a measurement system when appropriate reference material are not available. PE samples are equivalent to matrix spikes prepared by a third party that undergo certification analysis and can be non-blind, single-blind, or double-blind.

- Matrix Spike Samples. Matrix spike samples are environmental samples that are spiked in the laboratory with a known concentration of a target analyte(s) to verify percent recoveries. They are used primarily to check sample matrix interferences but can also be used to monitor laboratory performance. However, a data set of at least three or more

results is necessary to distinguish between laboratory performance and matrix interference (EPA 1991g). Matrix Spike samples are often replicated to monitor method performance and evaluate error due to laboratory bias and precision (when four or more pairs are analyzed). These replicates are often collectively referred to as a matrix spike/matrix spike duplicate (MS/MSD).

There are several additional terms applied to samples prepared by adding a known amount of the radionuclide of interest to the sample. The majority of these samples are designed to isolate individual sources of bias within a measurement system by preparing pre- and post-operation spikes. For example, the bias from the digestion phase of the measurement system can be determined by comparing the result from a pre-digest spike to the result from a post-digest spike.

There are also several types of samples used to estimate bias caused by contamination.

- Background Sample. A background sample is a sample collected upgradient of the area of potential contamination (either onsite or offsite) where there is little or no chance of migration of the contaminants of concern (EPA 1991g). Background samples are collected from the background reference area (Section 4.5), determine the natural composition and variability of the soil (especially important in areas with high concentrations of naturally occurring radionuclides), and are considered "clean" samples. They provide a basis for comparison of contaminant concentration levels with samples collected from the survey unit when the statistical tests described in Chapter 8 are performed.

- Field Blanks. Field blanks are samples prepared in the field using certified clean sand or soil and then submitted to the laboratory for analysis (EPA 1991g). A field blank is used to evaluate contamination error associated with sampling methodology and laboratory procedures. It also provides information about contaminants that may be introduced during sample collection, storage, and, transport. Field blanks are recommended for determining bias resulting from contamination for a radiation survey or site investigation.

- Method Blank. A method blank is an analytical control sample used to demonstrate that reported analytical results are not the result of laboratory contamination (ATSDR 1992). It contains distilled or deionized water and reagents, and is carried through the entire analytical procedure (laboratory sample preparation, digestion, and analysis). The method blank is also referred to as a reagent blank. The method blank is generally used as an internal control tool by the laboratory because it is a non-blind sample.

7.2.2.3 Representativeness

Representativeness is a measure of the degree to which data accurately and precisely represent a characteristic of a population parameter at a sampling point (ASQC 1995). Representativeness is a qualitative term that is reflected in the survey design through the selection of a measurement method (*e.g.*, direct measurement or sampling) and the size of a sample collected for analysis.

Sample collection and analysis is typically less representative of true radionuclide concentrations at a specific measurement location than performing a direct measurement. This is caused by the additional steps required in collecting and analyzing samples, such as sample collection, field sample preparation, laboratory sample preparation, and radiochemical analysis. However, direct measurement techniques with acceptable detection limits are not always available. When sampling is required as part of a survey design, it is critical that the sample collection procedures consider representativeness. The location of the sample is determined in Section 5.5.2.5, but the size and content of the sample are usually determined as the sample is collected. Sample size and content are discussed in Section 4.7.3 and Section 7.5. Sample collection procedures also need to consider the development of the DCGLs when determining the representativeness of the samples.

7.2.2.4 Comparability

Comparability is a qualitative term that expresses the confidence that two data sets can contribute to a common analysis and interpolation. Generally, comparability is provided by using the same measurement system for all analyses of a specific radionuclide. In many cases, equivalent procedures used within a measurement system are acceptable. For example, using a liquid-liquid extraction purification step to determine the concentration of ^{238}Pu using alpha spectrometry may be equivalent to using an ion-exchange column purification step. However, using a gross alpha measurement on a gas proportional counting system would not be considered equivalent. Comparability is usually not an issue except in cases where historical data have been collected and are being compared to current analytical results, or when multiple laboratories are used to provide results as part of a single survey design.

7.2.2.5 Completeness

Completeness is a measure of the amount of valid data obtained from the measurement system, expressed as a percentage of the number of valid measurements that should have been collected. Completeness is of greater concern for laboratory analyses than for direct measurements because the consequences of incomplete data often require the collection of additional samples. Direct measurements can usually be repeated fairly easily. The collection of additional samples generally requires a remobilization of sample collection personnel which can be expensive. Conditions at the site may have changed making it difficult or impossible to collect

representative and comparable samples without repeating the entire survey. On the other hand, if it is simply an analytical problem and sufficient sample was originally collected, the analysis can be repeated using archived sample material. Samples collected on a grid to locate areas of elevated activity are also a concern for completeness. If one sample analysis is not valid, the entire survey design for locating areas of elevated activity may be invalidated.

7.2.2.6 Other Data Quality Indicators

Several additional data quality indicators that influence the final status survey design are identified as DQOs in Section 2.3.1. Many of these (*e.g.*, selection and classification of survey units, decision error rates, variability in the contaminant concentration, lower bound of the gray region) are used to determine the number of measurements and are discussed in detail in Section 5.5. The method detection limit is directly related to the selection of a measurement method and a radionuclide-specific analytical technique.

Analytical methods should be capable of measuring levels below the established DCGLs, detection limits of 10-50% of the DCGL should be the target (see Section 6.7). Cost, time, best available technology, or other constraints may create situations where the above stated sensitivities are deemed impracticable. Under these circumstances, higher detection sensitivities may be acceptable. Although laboratories will state detection limits, these sensitivities are usually based on ideal or optimistic situations and may not be achievable under actual measurement conditions. Detection limits are subject to variation from sample to sample, instrument to instrument, and procedure to procedure, depending on sample size, geometry, background, instrument efficiency, chemical recovery, abundance of the radiations being measured, counting time, self-absorption in the prepared sample, and interferences from radionuclides or other materials present in the sample. The detection limit that is achievable in practice should not exceed the DCGL.

7.3 Communications with the Laboratory

Laboratory analyses of samples are generally performed by personnel not directly involved in the collection of the samples being analyzed. Samples are typically collected by one group working in the field, and analyzed by a second group located in a laboratory. This separation of tasks can potentially lead to problems based on the lack of communication between the two groups. For this reason, communications between the Project Manager, field personnel, and laboratory personnel are vital to ensuring the success of a project.

7.3.1 Communications During Survey Planning

The radioanalytical laboratory is a valuable resource during survey planning. Information on available analytical techniques, analytical bias and precision, method detection limits, analytical costs, and turnaround times can easily be provided by the laboratory. All of this information is used to make the decision to perform direct measurements or collect samples for laboratory measurements. Additional information, such as required sample size/volume, type of sample container, preservative requirements, and shipping requirements, including the availability of the laboratory for receipt of samples on weekends or holidays, can be obtained and factored into the survey plan.

Involving the radioanalytical laboratory during survey planning also provides the laboratory with site-specific information about the project. Information on the radionuclides of interest, possible chemical and physical form of the contamination, and mechanism for release of the contamination to the environment is used to modify or develop the analytical method for site-specific conditions if required. The laboratory should also be provided with the site-specific action levels (i.e., DCGLs, investigation levels) early in the survey planning process.

In some cases, it is not practical to select a radioanalytical laboratory early in the survey process to participate in the survey planning activities. For example, Federal procurement procedures require that a statement of work (SOW) identifying the tasks to be performed by the laboratory be developed prior to selecting a laboratory. Unfortunately, the details of the tasks for the laboratory to perform are developed during survey planning. This means that the information provided by the laboratory and used during survey planning will be obtained from another source, usually a radiochemist or health physicist trained in radiochemistry. The uncertainty associated with this information and subsequent decisions made based on this information increases. This may lead to increased costs caused by specifying an unnecessarily expensive analytical method in the SOW or repeated sampling and analysis of samples that did not meet the target detection limits because the specified analytical method was not sensitive enough. In addition, unnecessary or inappropriate analytical methods may be selected by the laboratory because site-specific information concerning the samples was not provided.

The laboratory should be consulted when planning the schedule for the survey to insure that the expected turnaround times can be met based on the projected laboratory workload.

7.3.2 Communications Before and During Sample Collection

In most situations, the sample collection and shipping containers are supplied by the laboratory; therefore, the laboratory should be notified well in advance of the sampling trip so that these items will be available to the sampling team during the survey.

The main purpose of communications with the laboratory during sample collection is to inform the laboratory of modifications to the survey design specified in the planning documents (*e.g.*, QAPP and SOPs). The laboratory should have a copy of the survey design in their possession prior to samples being collected.

Modifications to the survey design are often minor deviations from the SOPs caused by site-specific conditions and usually affect a small number of samples. For example, a rock outcropping covered by a thin layer of soil may restrict the depth of the surface soil sample to 5 cm (2 in.) instead of the 10 cm (4 in.) specified in the SOP. The mass of the samples collected from this area of the site is one-half the expected sample mass, and the laboratory needs to be informed of this deviation from the SOP.

In other situations, there may be an extensive modification to the number or types of samples collected at the site that will affect the analytical methods, detection capabilities, analytical costs, or even the assumptions used to develop the DCGL. For example, a large portion of the site may have been converted to a parking lot. A large pile of material that may represent the former surface soil will be sampled as well as soil collected from beneath the parking lot surface. The number of samples to be analyzed has doubled compared to the original SOW.

If the expected timing of receipt of samples at the laboratory changes due to sample collection schedule deviations, the laboratory should be notified. Most laboratories require prior notification for samples to be received on weekends.

7.3.3 Communications During Sample Analysis

The laboratory should communicate with the Project Manager and field personnel during sample analysis. The laboratory should provide a list of missing or damaged samples as soon after the samples are received as practical. This allows the Project Manager to determine if resampling is required to replace the missing or damaged samples. The Project Manager may also request notification from the laboratory when samples are spilled or lost during analysis. Preliminary reports of analytical results may be useful to help direct sampling activities and provide early indications of whether the survey objectives defined by the DQOs are being met. However, if preliminary results have not been verified or validated, their usefulness is limited.

7.3.4 Communications Following Sample Analysis

Following sample analysis, the laboratory will provide documentation of the analytical results as specified in the survey design. Laboratory personnel should be available to assist with data verification and validation.

7.4 Selecting a Radioanalytical Laboratory

Once the decision to perform sampling activities is made, the next step is to select the analytical methods and determine the data needs for these methods. It is advisable to select a radiochemical laboratory early in the survey planning process in order that it may be consulted on the analytical methodology[1] and the sampling activities. In addition, mobile laboratories can provide on-site analytical capability. Obtaining laboratory or other services may involve a specific procurement process. Federal procurement procedures may require additional considerations beyond the method described here.

The procurement of laboratory services usually starts with the development of a request for proposal that includes a statement-of-work describing the analytical services to be procured. The careful preparation of the statement-of-work is essential to the selection of a laboratory capable of performing the required services in a technically competent and timely manner.

The technical proposals received in response to the procurement request for proposal must be reviewed by personal familiar with radioanalytical laboratory operations in order to select the most qualified offerer. For complicated sites with a large number of laboratory analyses, it is recommended that a portion of this evaluation take the form of a pre-award audit. The provision for this audit must be in the request for proposal. The results of this audit provide a written record of the decision to use a specific laboratory. Smaller sites or facilities may decide that a review of the laboratory's qualifications is sufficient for the evaluation.

There are six criteria that should be reviewed during this evaluation:

- Does the laboratory possess the appropriate well-documented procedures, instrumentation, and trained personnel to perform the necessary analyses? Necessary analyses are defined by the data needs (radionuclide(s) of interest and target detection limits) identified by the DQO process.

- Is the laboratory experienced in performing the same or similar analyses?

- Does the laboratory have satisfactory performance evaluation results from formal monitoring or accreditation programs? The laboratory should be able to provide a summary of QA audits and proof of participation in interlaboratory cross-check programs. Equipment calibrations should be performed using National Institute of Standards and Technology (NIST) traceable reference radionuclide standards whenever possible.

[1] The laboratory provides information on personnel, capabilities, and current workload that are necessary inputs to the decision-making process.

- Is there an adequate capacity to perform all analyses within the desired timeframe? This criterion considers whether or not the laboratory possesses a radioactive materials handling license or permit for the samples to be analyzed. Very large survey designs may indicate that more than one analytical laboratory is necessary to meet the survey objectives.[2]

- Does the laboratory provide an internal quality control review of all generated data that is independent of the data generators?

- Are there adequate protocols for method performance documentation and sample security?

Providers of radioanalytical services should have an active and fully documented QA program in place.[3] This program should comply with the objectives determined by the DQO process in Section 2.3. The QA program should include:

- laboratory organizational structure
- personnel qualifications
- written standard operating procedures and instructions
- inter- and intralaboratory performance analyses
- design control to define the flow of samples through the laboratory
- a corrective action plan
- an internal audit program

Chain-of-Custody requirements and numbers of samples are also specified. The analytical procedures as well as the documentation and reporting requirements should be specified and agreed upon. These topics are discussed in detail in the following sections of this chapter.

7.5 Sampling

This section provides guidance on developing appropriate sample collection procedures for surveys designed to demonstrate compliance with a dose- or risk-based regulation. Sample collection procedures are concerned mainly with ensuring that a sample is representative of the sample media, is large enough to provide sufficient material to achieve the desired detection limit, and is consistent with assumptions used to develop the conceptual site model and the DCGLs. Additional considerations for sample collection activities are discussed in Section 4.7.3.

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[2] If several laboratories are performing analyses as part of the survey, the analytical methods used to perform the analyses should be similar to ensure comparability of results (see Appendix N, Section N.6.5).

[3] The QA program is typically documented in one or more documents such as a Quality Management Plan, Quality Assurance Manual, or Quality Assurance Project Plan.

The presence of radioactive and hazardous chemical wastes (mixed wastes) at a site can influence the survey design. The external exposure rates or radioactivity concentration of a specific sample may limit the time that workers will be permitted to remain in intimate contact with the samples, or may dictate that smaller samples be taken and special holding areas be provided for collected samples prior to shipment. These special handling considerations may conflict with the size specifications for the analytical method, normal sampling procedures, or equipment. There is a potential for biasing sampling programs by selecting samples that can be safely handled or legally shipped to support laboratories. Because final status surveys are performed to demonstrate that a site can be safely released, issues associated with high levels of radioactivity are not expected to be a concern.

7.5.1 Surface Soil

The purpose of surface soil sampling is to collect samples that accurately and precisely represent the radionuclides and their concentrations at the location being sampled. In order to do this and plan for sampling, a decision must be made as to the survey design. The selection of a survey design is based on the Historical Site Assessment, results from preliminary surveys (*i.e.*, scoping characterization, remedial action support), and the objectives of the survey developed using the Data Quality Objectives (DQO) Process. The selection between judgmental, random, and systematic survey designs is discussed in Section 5.5.3.

7.5.1.1 Sample Volume

The volume of soil collected should be specified in the sample collection procedure. In general, large volumes of soil are more representative than small volumes of soil. In addition, large samples provide sufficient sample to ensure that required detection limits can be achieved and that sample reanalysis can be done if there is a problem. However, large samples may cause problems with shipping, storage, and disposal. All of these issues should be discussed with the sample collection team and the analytical laboratory during development of sample collection procedures. In general, surface soil samples range in size from 100 g up to several kilograms.

The sample collection procedure should also make clear if it is more important to meet the volume requirement of the survey design or the surface area the sample represents. Constant volume is related to comparability of the results while surface area is more closely related to the representativeness of the results. Maintaining a constant surface area and depth for samples collected for a particular survey can eliminate problems associated with different depth profiles. The actual surface area included as part of the sample may be important for estimating the probability of locating areas of elevated concentration.

7.5.1.2 Sample Content

The material present in the field at the sample location may or may not provide a representative sample. Vegetative cover, soil particle size distribution, inaccessibility, or lack of sample material are examples of problems that may be identified during sample collection. All deviations from the survey design as documented in the Standard Operating Procedures (SOPs) should be recorded as part of the field sample documentation.

Sample content is generally defined by the assumptions used to develop the conceptual site model and the DCGLs. A typical agricultural scenario assumes that the top few centimeters of soil are available for resuspension in air, that the top 15 cm (6 in.) are homogenized by agricultural activities (*e.g.*, plowing), that roots can extend down several meters to obtain water and nutrients depending on the plant, and that external exposure is based on an assumed thickness of contaminated soil (usually at the surface). Depending on the dominant exposure pathways for each radionuclide, this can result in a complicated set of instructions for collecting representative samples. This situation can be further complicated by the fact that the site is not currently being used for agricultural purposes. For this situation it is necessary to look at the analytical results from the preliminary surveys (*i.e.*, scoping, characterization, remedial action support) to determine the expected depth of contamination.

In most situations the vegetative cover is not considered part of the surface soil sample and is removed in the field. For agricultural scenarios where external exposure is not the primary concern, soil particles greater than 2 mm (0.08 in.) are generally not considered as part of the sample (EPA 1990). Foreign material (*e.g.*, plant roots, glass, metal, or concrete) is also generally not considered part of the sample, but should be reviewed on a site-specific basis. It is important that the sample collection procedure clearly indicate what is and what is not considered part of the sample.

7.5.1.3 Sampling Equipment

The selection of proper sampling equipment is important to ensure that samples are collected effectively and efficiently. Sampling equipment generally consists of a tool to collect the sample and a container to place the collected sample in. Sample tracking begins as soon as the sample is collected, so it may be necessary to consider security of collected samples required by the objectives of the survey.

Sampling tools are selected based on the type of soil, sample depth, number of samples required, and training of available personnel. The selection of a sampling tool may also be based on the expected use of the results. For example, if a soil sample is collected to verify the depth profile used to develop the calibration for *in situ* gamma spectrometry, it is important to preserve the soil core. Table 7.1 lists several examples of tools used for collecting soil samples, situations where they are applicable, and some advantages and disadvantages involved in their use.

Table 7.1 Soil Sampling Equipment*

Equipment	Application	Advantages/Disadvantages
Tier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stone or dry soil.
Scoop or trowel	Soft surface soil	Inexpensive; easy to use and decontaminate; trowels with painted surfaces should be avoided
Bulb Planter	Soft Soil, 0-15 cm (0-6 in.)	Easy to use and decontaminate: uniform diameter and sample volume; preserves soil core; limited depth capability; can be difficult to decontaminate
Soil Coring Device	Soft soil, 0-60 cm (0-24 in.)	Relatively easy to use; preserves soil core; limited depth capability; can be difficult to decontaminate
Thin-wall tube sampler	Soft soil, 0-3 m (0-10 ft)	easy to use; preserves soil core; easy to decontaminate; can be difficult to remove cores
Split spoon sampler	Soil, to bedrock	Excellent depth range; preserves soil core; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores
Shelby tube sampler	Soft soil, to bedrock	Excellent depth range; preserves soil core; tube may be used for shipping core to lab.; may be used in conjunction with drill rig for obtaining deep cores
Bucket auger	Soft soil, 7.5 cm - 3 m (3 in. - 10 ft)	Easy to use; good depth range; uniform diameter and sample volume; may disrupt and mix soil horizons greater than 15 cm
Hand -operated power auger	Soil, 15 cm - 4.5 m (6 in. -15 ft)	Good depth range; generally used in conjunction with bucket auger; destroys soil core; requires two or more operators; can be difficult to decontaminate

* Reproduced from EPA 1991g

Sample containers are generally not a major concern for collecting surface soil samples. Polyethylene bottles with screw caps and wide mouths are recommended. These containers are fairly economical, provide easy access for adding and removing samples, and resist chemicals, breaking, and temperature extremes. Glass containers are also acceptable, but they are fragile and tend to break during shipment. Metal containers are sometimes used, but sealing the container can present a problem and corrosion can be an issue if the samples are stored for a significant length of time.

7.5.2 Building Surfaces

Because building surfaces tend to be relatively smooth and the radioactivity is assumed to be on or near the surface, direct measurements are typically used to provide information on contaminant concentrations. Sometimes, however, it is necessary to collect actual samples of the building material surface for analysis in a laboratory.

7.5.2.1 Sample Volume

The sample volume collected from building surfaces is usually a less significant DQO concern than the area from which the sample was collected. This is because building surface DCGLs are usually expressed in terms of activity per unit area. It is still necessary to consider the sample volume to account for sample matrix effects that may reduce the chemical recovery, which in turn has an affect on the detection limit.

7.5.2.2 Sample Content

If residual activity is covered by paint or some other treatment, the underlying surface and the coating itself may be contaminated. If the activity is a pure alpha or low-energy beta emitter, measurements at the surface will probably not be representative of the actual residual activity level. In this case the surface layer is removed from the known area, such as by using a commercial stripping agent or by physically abrading the surface. The removed coating material is analyzed for activity content and the level converted to appropriate units (*i.e.*, Bq/m^2, $dpm/100\ cm^2$) for comparison with surface activity DCGLs. Direct measurements can be performed on the underlying surface after removal of the coating.

Residual radioactivity may be incorporated into building materials, such as pieces of concrete or other unusual matrices. Development of SOPs for collecting these types of samples may involve consultation with the analytical laboratory to help ensure that the objectives of the survey are achieved.

The thickness of the layer of building surface to be removed as a sample should be consistent with the development of the conceptual site model and the DCGLs. For most sites the surface layer will only be the first few millimeters of the material being sampled.

7.5.2.3 Sampling Equipment

Tools used to provide samples of building surfaces depend on the material to be sampled. Concrete may require chisels, hammers, drills, or other tools specifically designed to remove a thin layer of the surface. Wood surfaces may require using a sander or a saw to collect a sample. Paint may be chemically or physically stripped from the surface.

Sample containers for these samples are generally the same as those recommended for soil samples. If chemicals are used to strip paint or other surface materials, the chemical resistance of the container should be considered.

7.5.3 Other Media

Surface soil and building surfaces are the media addressed in MARSSIM during the final status survey design. Other media may be involved and may have been remediated. Data collection activities during preliminary surveys (*i.e.*, scoping, characterization, remedial action support) may involve collecting samples of other media to support the final status survey design. Examples of other media that may be sampled include:

- subsurface soil
- ground water
- surface water
- sediments
- sewers and septic systems
- flora and fauna (plants and animals)
- airborne particulates
- air (gas)

Appendix M provides a list of resources that can be used to develop sample collection procedures for other media that may required by preliminary surveys to support the development of a final status survey design.

7.6 Field Sample Preparation and Preservation

Proper sample preparation and preservation are essential parts of any radioactivity sampling program. The sampling objectives should be specified before sampling activities begin. Precise records of sample collection and handling are necessary to ensure that data obtained from different locations or time frames are correctly compared.

The appropriateness of sample preparation techniques is a function of the analysis to be performed (EPA 1992a, 1992b). Field sample preparation procedures are a function of the specified analysis and the objectives of the survey. It is essential that these objectives be clearly established and agreed upon in the early stages of survey planning (see Section 2.3).

7.6.1 Surface Soil

Soil and sediment samples, in most protocols, require no field preparation and are not preserved. In some protocols, cooling of soil samples to 4 ^{o}C is required during shipping and storage of soil samples. This is not a practice normally followed for the radiochemical analysis of soil samples.

When replicate samples are prepared in the field, it is necessary to homogenize the sample prior to separation into replicates. There are standard procedures for homogenizing soil in the laboratory (ASTM 1995), but the equipment required for these procedures may not be available in the field. Simple field techniques, such as cone and quarter, or using a riffle splitter to divide the sample may be appropriate if the sample can be dried (ASTM 1993, EPA 1991g). If the sample contains significant amounts of residual water (*e.g.*, forms clumps of soil) and there are no facilities for drying the sample, it is recommended that the homogenization and separation into replicates be performed in a laboratory. It is preferable to use non-blind replicates where the same laboratory prepares and analyzes the replicates rather than use poorly homogenized or heterogeneous samples to prepare replicates samples.

7.6.2 Building Surfaces

Field preparation and preservation of building and associated materials, including smear samples, is not generally required. Homogenization of samples to prepare replicates is the same for building surface material and soil.

7.6.3 Other Media

Other media may have significant requirements related to field sample preparation and preservation. For example, water samples may need filtering and acidification. Storage at reduced temperatures (*i.e.*, cooling or freezing) to reduce biological activity may be necessary for some samples. Addition of chemical preservatives for specific radionuclides or media may also be required.

7.7 Analytical Procedures

The selection of the appropriate radioanalytical methods is normally made prior to the procurement of analytical services and is included in the statement-of-work of the request for proposal. The statement-of-work may dictate the use of specific methods or be performance based. Unless there is a regulatory requirement, such as conformance to the EPA drinking water methods (EPA 1980a), the specification of performance based methodology is encouraged. One reason for this is that a laboratory will usually perform better using the methods routinely employed in its laboratory as contrasted to using other methods with which it has less experience.

The laboratory is also likely to have historical data on performance for methods routinely used by that laboratory. However, the methods employed in a laboratory should be derived from a reliable source, such as those listed in Table 7.2.

Table 7.2 Examples of References for Routine Analytical Methods

- *Methods of Air Sampling and Analysis* (Lodge 1988)
- *Annual Book of ASTM Standards, Water and Environmental technology. Volume 11.04, Environmental Assessment; Hazardous Substances and Oil Spill Responses; Waste Management; Environmental Risk Assessment* (ASTM 1997)
- *Standard Methods for the Examination of Water and Wastewater* (APHA 1995)
- *EML Procedures Manual* (DOE 1990b)
- *Radiochemical Analytical Procedures for Analysis of Environmental Samples* (EPA 1979)
- *Radiochemistry Procedures Manual* (EPA 1984a)
- *Indoor Radon and Radon Decay Product Measurement Protocols* (EPA 1992d)
- *USAEHA Environmental Sampling Guide* (Department of the Army 1993)

This section briefly describes specific equipment and procedures to be used once the sample is prepared for analysis. The results of these analyses (*i.e.*, the levels of radioactivity found in these samples) are the values used to determine the level of residual activity at a site. In a decommissioning effort, the DCGLs are expressed in terms of the concentrations of certain radionuclides. It is of vital importance, therefore, that the analyses be accurate and of adequate sensitivity for the radionuclides of concern. The selection of analytical procedures should be coordinated with the laboratory and specified in the survey plan.

Analytical methods should be adequate to meet the data needs identified in the DQO process. Consultation with the laboratory performing the analysis is recommended before selecting a course of action. MARSSIM is not intended to limit the selection of analytical procedures, rather all applicable methods should be reviewed to provide results that meet the objectives of the survey. The decision maker and survey planning team should decide whether routine methods will be used at the site or if non-routine methods may be acceptable.

- Routine analytical methods are documented with information on minimum performance characteristics, such as detection limit, precision and accuracy, and useful range of radionuclide concentrations and sample sizes. Routine methods may be issued by a recognized organization (*e.g.*, Federal or State agency, professional organization), published in a refereed journal, or developed by an individual laboratory. Table 7.2 lists examples of sources for routine methods.

- Non-routine methods address situations with unusual or problematic matrices, low detection limits, or new parameters, procedures or techniques. Non-routine methods include adjustments to routine methods, new techniques published in refereed literature, and development of new methods.

References that provide information on radiochemical methodology and should be considered in the methods review and selection process are available from such organizations as:

- National Council on Radiation Protection and Measurements (NCRP)
- American Society of Testing and Materials (ASTM)
- Radiological and Environmental Sciences Laboratory (RESL), Idaho Falls, Idaho (Operated by the DOE)
- DOE Technical Measurements Center, Grand Junction, CO
- Environmental Measurements Laboratory (EML); formerly the Health and Safety Laboratory of the DOE

Equipment vendor literature, catalogs, and instrument manuals are often a source of useful information on the characteristics of radiation detection equipment. Table 7.3 provides a summary of common laboratory methods with estimated detection limits.

Analytical procedures in the laboratory consist of several parts that are assembled to produce an SOP for a specific project or sample type. These parts include:

- laboratory sample preparation
- sample dissolution
- sample purification
- preparation for counting
- counting
- data reduction

Table 7.3 Typical Measurement Sensitivities for Laboratory Radiometric Procedures

Sample Type	Radionuclides or Radiation Measured	Procedure	Approximate Measurement Sensitivity
Smears (filter paper)	Gross alpha	Gas-flow proportional counter; 5-min count Alpha scintillation detector with scaler; 5-min count	5 dpm 20 dpm
	Gross beta	Gas-flow proportional counter; 5-min count End window GM with scaler; 5-min count (unshielded detector)	10 dpm 80 dpm
	Low energy beta (^{3}H, ^{14}C, ^{63}Ni)	Liquid scintillation spectrometer; 5-min count	30 dpm
Soil Sediment	^{137}Cs, ^{60}Co, ^{226}Ra (^{214}Bi)[a], ^{232}Th (^{228}Ac), ^{235}U	Germanium detector (25% relative efficiency) with multichannel analyzer; pulse height analyzer; 500-g sample; 15-min analysis	0.04-0.1 Bq/g (1-3 pCi/g)
	$^{234, 235, 238}$U; $^{238, 239, 240}$Pu; $^{227, 228, 230, 232}$Th; other alpha emitters	Alpha spectroscopy with multichannel analyzer - pyrosulfate fusion and solvent extraction; surface barrier detector; pulse height analyzer; 1-g sample; 16-hr count	0.004-0.02 Bq/g (0.1-0.5 pCi/g)
Water	Gross alpha	Gas-flow proportional counter; 100-ml sample, 200-min count	0.04 Bq/L (1 pCi/l)
	Gross beta	Gas-flow proportional counter; 100-ml sample, 200-min count	0.04 Bq/L (1 pCi/L)
	^{137}Cs, ^{60}Co, ^{226}Ra (^{214}Bi), ^{232}Th (^{228}Ac), ^{235}U	Germanium detector (25% relative efficiency) with multichannel analyzer; pulse height analyzer; 3.5L sample, 16-hr count	0.4 Bq/L (10 pCi/L)
	$^{234, 235, 238}$U; $^{238, 239, 240}$Pu; $^{227, 228, 230, 232}$Th; other alpha emitters	Alpha spectroscopy with multichannel analyzer - solvent extraction; surface barrier detector; pulse height analyzer; 100 ml sample, 30 min count	0.004-0.02 Bq/L (0.1-0.5 pCi/L)
	^{3}H	Liquid scintillation spectrometry; 5-ml sample, 30-min count	10 Bq/L (300 pCi/L)

[a] Indicates that a member of the decay series is measured to determine activity level of the parent radionuclide of primary interest.

7.7.1 Photon Emitting Radionuclides

There is no special sample preparation required for counting samples using a germanium detector or a sodium iodide detector beyond placing the sample in a known geometry for which the detector has been calibrated. The samples can be measured as they arrive at the laboratory, or the sample can be dried, ground to a uniform particle size, and mixed to provide a more homogeneous sample if required by the SOPs.

The samples are typically counted using a germanium detector with a multichannel analyzer or a sodium iodide detector with a multichannel analyzer. Germanium detectors have better resolution and can identify peaks (and the associated radionuclides) at lower concentrations. Sodium iodide detectors often have a higher efficiency and are significantly less expensive than germanium detectors. Low-energy photons (*i.e.*, x-rays and gamma rays below 50 keV) can be measured using specially designed detectors with an entrance window made from a very light metal, typically beryllium. Descriptions of germanium and sodium iodide detectors are provided in Appendix H.

Data reduction is usually the critical step in measuring photon emitting radionuclides. There are often several hundred individual gamma ray energies detected within a single sample. Computer software is usually used to identify the peaks, associate them with the proper energy, associate the energy with one or more radionuclides, correct for the efficiency of the detector and the geometry of the sample, and provide results in terms of concentrations with the associated uncertainty. It is important that the software be either a well-documented commercial package or thoroughly evaluated and documented before use.

7.7.2 Beta Emitting Radionuclides

Laboratory sample preparation is an important step in the analysis of surface soil and other solid samples for beta emitting radionuclides. The laboratory will typically have a sample preparation procedure that involves drying the sample and grinding the soil so that all of the particles are less than a specified size to provide a homogeneous sample. A small portion of the homogenized sample is usually all that is required for the individual analysis.

Once the sample has been prepared, a small portion is dissolved, fused, or leached to provide a clear solution containing the radionuclide of interest. The only way to ensure that the sample is solubilized is to completely dissolve the sample. However, this can be an expensive and time-consuming step in the analysis. In some cases, leaching with strong acids can consistently provide greater than 80% recovery of the radionuclide of interest (NCRP 1976a) and may be acceptable for certain applications. Gross beta measurements may be performed on material that has not been dissolved.

After dissolution, the sample is purified using a variety of chemical reactions to remove bulk chemical and radionuclide impurities. The objective is to provide a chemically and radiologically pure sample for measurement. Examples of purification techniques include precipitation, liquid-liquid extraction, ion-exchange chromatography, distillation, and electrodeposition. Gross beta measurements may be performed on material that has not been purified.

After the sample is purified, it is prepared for counting. Beta emitting radionuclides are usually prepared for a specific type of counter in a specified geometry. Solid material is usually precipitated and collected on a filter in a circular geometry to provide a homogeneous sample. Liquid samples are typically converted to the appropriate chemical form and diluted to a specified volume in preparation for counting.

Measurements of solid samples are typically performed using a gas-flow proportional counter. Because total beta activity is measured, it is important that the purification step be performed to remove any interfering radionuclides. Liquid samples are usually diluted using a liquid scintillation cocktail and counted using a liquid scintillation spectrometer. Liquid scintillation spectrometers can be used for low-energy beta emitting radionuclides, such as ^{3}H and ^{63}Ni. They also have high counting efficiencies, but often have a high instrument background as well. Gas-flow proportional counters have a very low background. Appendix H provides a description of both the gas-flow proportional counter and the liquid scintillation spectrometer.

Data reduction for beta emitting radionuclides is less complicated than that for photon emitting radionuclides. Since the beta detectors report total beta activity, the calculation to determine the concentration for the radionuclide of interest is straightforward.

7.7.3 Alpha Emitting Radionuclides

Laboratory sample preparation for alpha emitting radionuclides is similar to that for beta emitting radionuclides. Sample dissolution and purification tasks are also similar to those performed for beta emitting radionuclides.

Because of the limited penetrating power of alpha particles, the preparation for counting is often a critical step. Gross alpha measurements can be made using small sample sizes with a gas-flow proportional counter, but self-absorption of the alpha particles results in a relatively high detection limit for this technique. Liquid scintillation spectrometers can also be used to measure alpha emitting radionuclides but the resolution limits the usefulness of this technique. Most alpha emitting radionuclides are measured in a vacuum (to limit absorption by air) using alpha spectroscopy. This method requires that the sample be prepared as a virtually weightless mount in a specific geometry. Electrodeposition is the traditional method for preparing samples for counting. This technique provides the highest resolution, but it requires a significant amount of

training and expertise on the part of the analyst to produce a high quality sample. Precipitation of the radionuclide of interest on the surface of a substrate is often used to prepare samples for alpha spectroscopy. While this technique generally produces a spectrum with lower resolution, the preparation time is relatively short compared to electrodeposition, and personnel can be trained to prepare acceptable samples relatively quickly.

Alpha emitting radionuclides are typically measured using alpha spectroscopy. The data reduction requirements for alpha spectroscopy are greater than those for beta emitting radionuclides, and similar to those for photon emitting radionuclides. Alpha spectroscopy produces a spectrum of alpha particles detected at different energies, but because the sample is purified prior to counting, all of the alpha particles come from radionuclides of a single element. This simplifies the process of associating each peak with a specific radionuclide, but the lower resolution associated with alpha spectroscopy increases the difficulty of identifying the peaks. Although commercial software packages are available for interpreting alpha spectroscopy results, an experienced operator is required to ensure that the software is working properly.

7.8 Sample Tracking

Sample tracking refers to the identification of samples, their location, and the individuals responsible for their custody and transfer of the custody. This process covers the entire process from collection of the samples and remains intact through the analysis and final holding or disposal. It begins with the taking of a sample where its identification and designation of the sample are critical to being able to relate the analytical result to a site location.

Tracking samples from collection to receipt at the analytical laboratory is normally done through a Chain of Custody process, and documented on a Chain-of-Custody (COC) record. Once samples are received by the laboratory, internal tracking (*e.g.*, COC) procedures should be in place and codified through SOPs that assure integrity of the samples. Documentation of changes in the custody of a sample(s) is important. This is especially true for samples that may be used as evidence to establish compliance with a release criterion. In such cases, there should be sufficient evidence to demonstrate that the integrity of the sample is not compromised from the time it is collected to the time it is analyzed. During this time, the sample should either be under the positive control of a responsible individual or secured and protected from any activity that could change the true value of the results or the nature of the sample. When this degree of sample handling or custody is necessary, written procedures should be developed for field operations and for interfacing between the field operations and the analytical laboratory. This ensures that a clear transfer of the custodial responsibility is well documented and no questions exist as to who is responsible for the sample at any time.

7.8.1 Field Tracking Considerations

- Field personnel are responsible for maintaining field logbooks with adequate information to relate the sample identifier (sample number) to its location and for recording other information necessary to adequately interpret results of sample analytical data.
- The sample collector is responsible for the care and custody of the samples until they are properly transferred or dispatched. This means that samples are in their possession, under constant observation, or secured. Samples may be secured in a sealed container, locked vehicle, locked room, *etc*.
- Sample labels should be completed for each sample using waterproof ink.
- The survey manager or designee determines whether or not proper custody procedures were followed during the field work, and decides if additional sampling is indicated.
- If photographs are included as part of the sampling documentation, the name of the photographer, date, time, site location, and site description should be entered sequentially in a logbook as the photos are taken. After the photographs are developed, the prints should be serially numbered.

7.8.2 Transfer of Custody

- All samples leaving the site should be accompanied by a Chain-of-Custody record. This record documents sample custody transfer from the sampler, often through another person, to the laboratory. The individuals relinquishing the samples should sign and date the record. The record should include a list, including sample designation (number), of the samples in the shipping container and the analysis requested for each sample.
- Shipping containers should be sealed and include a tamper indicating seal that will indicate if the container seal has been disturbed. The method of shipment, courier name, or other pertinent information should be listed in the Chain-of-Custody record.
- The original Chain-of-Custody record should accompany the samples. A copy of the record should be retained by the individual or organization relinquishing the samples.
- Discuss the custody objectives with the shipper to ensure that the objectives are met. For example, if the samples are sent by mail and the originator of the sample requires a record that the shipment was delivered, the package should be registered with return receipt requested. If, on the other hand, the objective is to simply provide a written record of the shipment, a certificate of mailing may be a less expensive and appropriate alternative.
- The individual receiving the samples should sign and date the record. The condition of the container and the tamper indicating seal should be noted on the Chain-of-Custody record. Any problems with the individual samples, such as a broken container, should be noted on the record.

7.8.3 Laboratory Tracking

When the samples are received by the laboratory they are prepared for radiochemical analyses. This includes the fractionation of the sample into aliquots. The tracking and Chain-of-Custody documentation within the laboratory become somewhat complicated due to the fact that several portions of the original sample may exist in the laboratory at a given time. The use of a computer based Laboratory Information System (LIMS) can greatly assist in tracking samples and fractions through the analytical system.

The minimal laboratory tracking process consists of the following:

- transfer of custody on receipt of the samples (original Chain-of-Custody form is retained by the laboratory and submitted with the data package for the samples)
- documentation of sample storage (location and amount)
- documentation of removal and return of sample aliquots (amount, date and time, person removing or returning, and reason for removal)
- transfer of the samples and residues to the receiving authority (usually the site from which they were taken)

The procedure for accomplishing the above varies from laboratory to laboratory, but the exact details of performing the operations of sample tracking should be contained in a SOP.

7.9 Packaging and Transporting Samples

All samples being shipped for radiochemical analysis should be properly packaged and labeled before transport offsite or within the site. The primary concern is the possibility of spills, leaks, or breakage of the sample containers. In addition to resulting in the loss of samples and cross-contamination, the possible release of hazardous material poses a threat to the safety of persons handling and transporting the package.

Suggestions on packaging and shipping radioactive environmental samples are listed below.

1) Review NRC requirements (10 CFR part 71) and Department of Transportation (DOT) requirements (49 CFR parts 170 through 189) for packaging and shipping radioactive environmental samples.

2) Visually inspect each sample container for indication of leaks or defects in the sample container.

a) Liquid samples should be shipped in plastic containers, if possible, and the caps on the containers should be secured with tape. One exception to the use of plastic bottles is samples collected for ^{3}H analyses which may require glass containers.

b) Heavy plastic bags, with sealable tops, can be used to contain solid samples (*e.g.*, soil, sediment, air filters). The zip-lock should be secured with tape. Heavy plastic lawn bags can be used to contain vegetation samples. The tops should be closed with a "tie" that is covered by tape to prevent it from loosening and slipping off.

3) Wipe individual sample containers with a damp cloth or paper towel to remove any exterior contamination. The outer surfaces of containers holding samples collected in a contaminated area should be surveyed with a hand-held instrument(s), appropriate for the suspected type of radioactivity (β/γ or α).

4) If glass sample containers are used, place sample containers inside individual plastic bags and seal in order to contain the sample in case of breakage.

5) Use packing material (*e.g.*, paper, styrofoam, "bubble wrap") to immobilize and isolate each sample container and buffer hard knocks on the outer container during shipping. This is especially important in cold weather when plastic containers may become brittle and water samples may freeze.

6) When liquid samples are shipped, include a sufficient quantity of an absorbent material (*e.g.*, vermiculite) to absorb all liquid packed in the shipping container in case of breakage. This absorbent material may suffice as the packing material described above in item 5.

7) Include the original, signed and dated, Chain-of-Custody (COC) form, identifying each sample in the package. It is good practice to place the COC form in a plastic bag to prevent it from becoming wet or contaminated in case of a spill during shipment. If possible, avoid having multiple packages of samples covered by a single COC form.

8) Seal closed the package and apply COC tape in such a manner that it must be torn (broken) in order to open the package. The tape should carry the signature of the sender, and the date and time, so that it cannot be removed and replaced undetected.

9) Ice chests, constructed of metal or hard plastic, make excellent shipping containers for radioactive environmental samples.

If samples are sent offsite for analysis, the shipper is responsible for complying with all applicable Federal, State, and local regulations. Applicable Federal regulations are briefly addressed below. Any State or local regulation will very likely reflect a Federal regulation.

7.9.1 U.S. Nuclear Regulatory Commission Regulations

NRC regulations for packaging, preparation, and shipment of licensed material are contained in 10 CFR Part 71: "Packaging and Transportation of Radioactive materials".

Samples containing low levels of radioactivity are exempted as set forth in §§ 71.10. A licensee is exempt from all requirements of Part 71 if the specific activity of the sample being shipped is not greater than 74,000 Bq/kg (2,000 pCi/g).

Low Specific Activity Material (LSAM) is defined in §§ 71.4: “Definitions.” Samples classified as LSAM need only meet the requirements of the U.S. Department of Transportation (DOT), discussed below, and the requirements of §§ 71.88: “Air transport of plutonium.” Most environmental samples will fall into this category.

7.9.2 U.S. Department of Transportation Regulations

The U.S. Department of Transportation provides regulations governing the transport of hazardous materials under the Hazardous Materials Transportation Act of 1975 (88 Stat. 2156, Public Law 93-633). Applicable requirements of the regulations are found in 49 CFR Parts 170 through 189. Shippers of samples containing radioactivity should be aware of the current rules in the following areas.

- Accident Reporting - 49 CFR 171
- Marking and Labeling Packages for Shipment - 49 CFR 172
- Packaging - 49 CFR 173
- Placarding a Package - 49 CFR 172
- Registration of Shipper/Carrier - 49 CFR 107
- Shipper Required Training - 49 CFR 172
- Shipping Papers & Emergency Information - 49 CFR 172
- Transport by Air - 49 CFR 175

- Transport by Rail - 49 CFR 174
- Transport by Vessel - 49 CFR 176
- Transport on Public Highway - 49 CFR 177

7.9.3 U.S. Postal Service Regulations

Any package containing radioactive materials is nonmailable if required to bear the U.S. Department of Transportation's Radioactive White-1 (49 CFR 172.436), Radioactive Yellow-II (49 CFR 172.438), or Radioactive Yellow-III (49 CFR 172.440) label, or if it contains quantities of radioactive material in excess of those authorized in Publication 6, Radioactive Material, of the U.S. Postal Service.