

11 SAMPLE RECEIPT, INSPECTION, AND TRACKING

11.1 Introduction

This chapter provides guidance on laboratory sample receiving and surveying, inspecting, documenting custody, and assigning laboratory tracking identifiers (IDs). These topics are presented sequentially in this chapter, but they may be performed in a different order. The chapter is directed primarily at laboratory personnel (as are all of the Part II chapters), although the project manager and field personnel need to be aware of the steps involved in sample receipt, inspection, and tracking. Within MARLAP, the “sample receipt” process includes the surveying of the package and sample containers for radiological contamination and radiation levels. “Sample inspection” means checking the physical integrity of the package and samples, confirming the identity of the sample, confirming field preservation (if necessary), and recording and communicating the presence of hazardous materials. “Laboratory sample tracking” is a process starting with logging in the sample and assigning a unique laboratory tracking identifier (numbers and/or letters) to be used to account for the sample through analyses, storage, and shipment. Laboratory tracking continues the tracking that was initiated in the field during sample collection (see Section 10.2, “Field Sampling Plan: Non-Matrix-Specific Issues”).

This chapter focuses on sample receipt, inspection, and tracking of samples in the laboratory because these are the three modes of initial control and accountability (Figure 11.1). Sample receipt and inspection activities need to be done in a timely manner to allow the laboratory and field personnel to resolve any problems (e.g., insufficient material collected, lack of field preservation, etc.) with the samples received by the laboratory as soon as is practical. Effective communications between field personnel and the laboratory not only facilitates problem resolution but also prevents unnecessary delays in the analytical process.

Other relevant issues, including the laboratory’s radioactive materials license conditions and proper operating procedures, are also discussed because these topics are linked to receipt, inspection, and tracking activities. The result of the sample receipt and inspection activities is to accept the samples as received or to perform the necessary corrective action (which may include rejecting samples). Health and safety information on radiological issues can be found in NRC (1998a; 1998b).

11.2 General Considerations

11.2.1 Communication Before Sample Receipt

Before the samples are received, the laboratory should know the approximate number of samples that will be received within a specific

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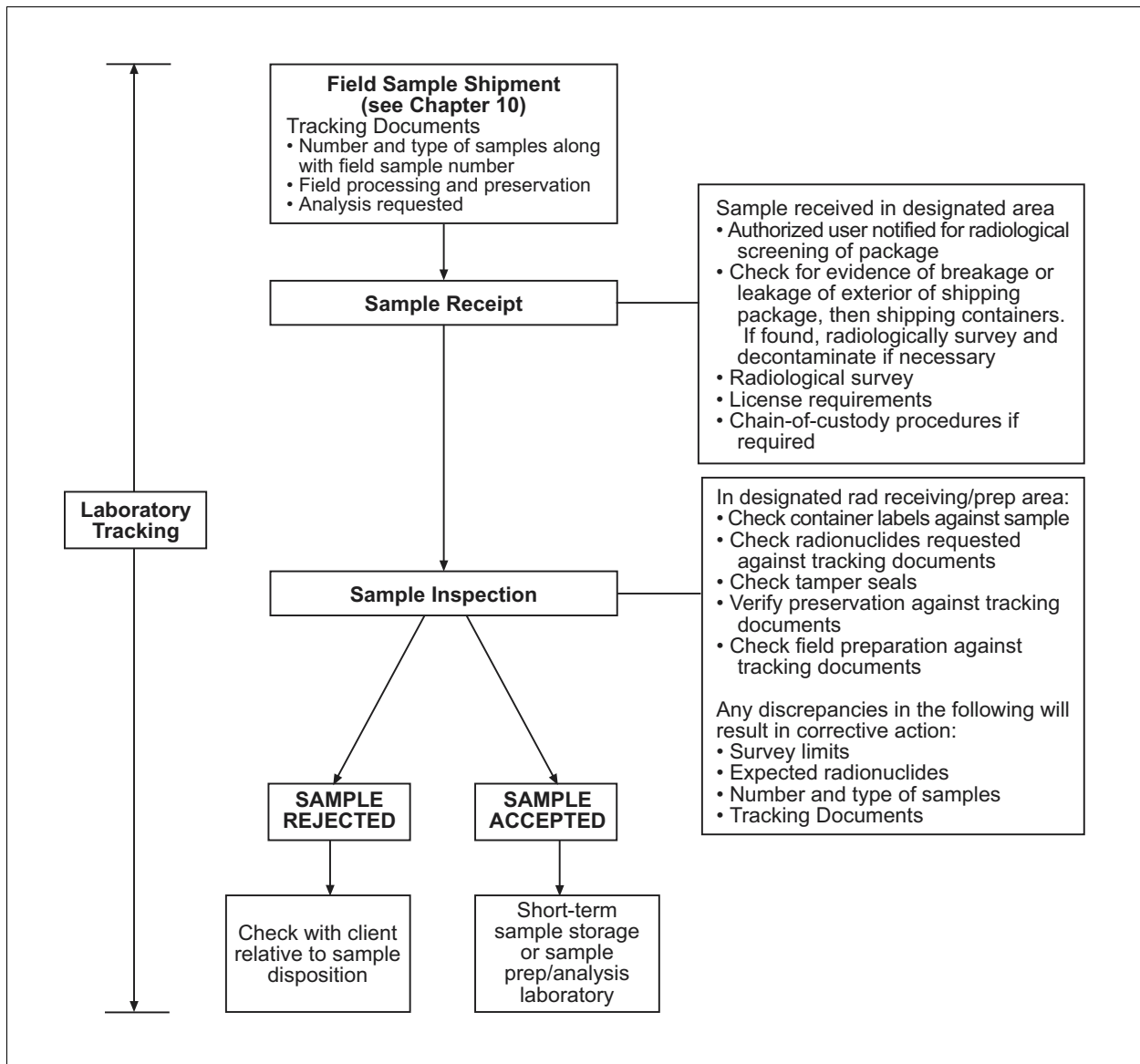


FIGURE 11.1 — Overview of sample receipt, inspection, and tracking

period of time and the types of analyses that are expected for the samples. Laboratory personnel should be provided with a contact in the field and with means of contacting the person (telephone, FAX, e-mail). The information about the client, points of contact, number of samples, and types of analyses can be entered into the laboratory information management system (LIMS) to facilitate communication between the laboratory—in both the sample receipt area and the project management area—and the project manager. Communication between laboratory personnel and project staff in the field allows the parties to coordinate activities, schedules, and sample receipt. In particular, the project manager should provide to the laboratory any special instructions regarding the samples before shipment of samples. This information serves to notify

the laboratory of health and safety concerns and provides details that will affect analytical procedures, sample disposition, etc. For example, without this communication, a laboratory might receive a partial shipment and not realize that samples are missing. Furthermore, advance communications allow laboratory staff to arrange for special handling or extra storage space should the need arise.

Planning for the samples to be received at the laboratory starts during the development of the appropriate plan document and the statement of work (SOW) and continues through the communication between the project staff in the field and the laboratory. For example, the laboratory could use its LIMS to generate labels and bar-codes for the appropriate containers to be used in the field. This process would assist in assigning appropriate sample IDs for the laboratory tracking system, which starts with sample receipt. The laboratory should instruct the field staff to place the tracking documents on the inside of the cooler lid for easy access and to include any other pertinent information (field documentation, field surveying information, etc.).

11.2.2 Standard Operating Procedures

A laboratory should have standard operating procedures (SOPs) for activities related to sample receipt, inspection, and tracking. Some typical topics that might be addressed in laboratory SOPs are presented in Table 11.1. For example, the laboratory should have an SOP that describes what information should be included in the laboratory sample tracking system. Laboratory SOPs should describe chain-of-custody procedures giving a comprehensive list of the elements in the program such as signing the appropriate custody forms, storing samples in a secure area, etc. (ASTM D4840; ASTM D5172; EPA, 1995).

TABLE 11.1 — Typical topics addressed in standard operating procedures related to sample receipt, inspection, and tracking

Sample	• Order and details for activities associated with receiving shipments of samples
Receipt:	• Surveying methods
Inspection:	<ul style="list-style-type: none"> • Check physical integrity • Confirm sample identification • Identify/manage hazardous materials • pH measurement instructions • Use the laboratory information management system (LIMS) to assign laboratory sample IDs
Tracking:	<ul style="list-style-type: none"> • Maintain chain of custody and document sample handling during transfer from the field to the laboratory, then within the laboratory • Ensure proper identification of samples throughout process • Procedures to quickly determine location and status of samples within laboratory
Custodian:	• Execution of responsibilities of the sample custodian
Forms/Labels:	• Examples of forms and labels used to maintain sample custody and document sample handling in the laboratory

The laboratory needs to establish corrective action guidelines (Section 11.3.3) as part of every SOP for those instances when a nonconformance is noted. Early recognition of a nonconformance will allow the project manager and the laboratory more options for a quick resolution.

11.2.3 Laboratory License

Laboratories that handle radioactive materials are required (with few exceptions, such as certain U.S. Department of Energy National Laboratories and Department of Defense laboratories) to have a radioactive materials license issued by the NRC or the Agreement State in which the laboratory operates. The radioactive materials license lists the radionuclides that the laboratory can possess, handle, and store. In addition, the license limits the total activity of specific radionuclides that can be in the possession of the laboratory at a given time.

The client must have a copy of the current radioactive materials license for the facility to which the samples are being shipped. The laboratory staff and the project manager all need to be aware of the type of radionuclide(s) in the samples and the total number of samples to be sent to the laboratory. This information should be included in the appropriate plan document and SOW prior to sampling.

The laboratory is required by the license to maintain a current inventory of certain radioactive materials present in the facility. The radioactive materials license also requires the laboratory to develop and maintain a radiation protection plan (NRC, 1998b) that states how radioactive samples will be received, stored, and disposed. The laboratory will designate an authorized user (NRC, 1998b) to receive the samples. A Radiation Safety Officer (RSO) may be an authorized user, but not always. NRC (1998b) gives procedures for the receipt of radioactive samples during working hours and non-working hours.

11.2.4 Sample Chain-of-Custody

Sample chain-of-custody (COC) is defined as a process whereby a sample is maintained under physical possession or control during its entire life cycle, that is, from collection to disposal (ASTM D4840—see Section 10.2.7). The purpose of COC is to ensure the security of the sample throughout the process. COC procedures dictate the documentation needed to demonstrate that COC is maintained. When a sample is accepted by the laboratory it is said to be in the physical possession or control of the laboratory. ASTM D4840 states that a sample is under “custody” if it is in possession or under control so as to prevent tampering or alteration of its characteristics.

If the samples are transferred under COC, the relinquisher and the receiver should sign the appropriate parts of the COC form with the date and time of transfer (see Figure 10.1). After receipt and inspection the samples should be kept in a locked area or in an area with controlled access.

COC is not a requirement for all samples. COC is most often required when the sample data may be used as legal evidence. The project plan should state whether COC will be required. The paperwork received with the samples should also indicate whether COC has been maintained from the time of collection and must be maintained in the laboratory. If the laboratory has been informed that COC procedures should be followed, but it appears that appropriate COC procedures have not been followed (before or after sample receipt at the laboratory) or there are signs of possible sample tampering when the samples arrive, the project manager should be contacted. The problem and resolution should be documented. Additional information on COC can be found in EPA (1985).

11.3 Sample Receipt

Laboratory sample receipt occurs when a package containing samples is accepted, the package and sample containers are surveyed for external surface radiological contamination and radiation level, and the physical integrity of the package and samples is checked. Packages include the shipping parcel that holds the smaller sample containers with the individual samples (see Section 11.3.2 on radiological surveying). Also note that topics and activities covered in Section 11.3 appear in a sequence but, in many cases, these activities are performed simultaneously during initial receiving activities (i.e., package surveying and observation of its physical integrity).

11.3.1 Package Receipt

Some laboratories require arriving samples to go through a security inspection process at a central receiving area before routing them to the appropriate laboratory area(s). In addition, if samples are shipped by an air transport carrier, the shipping containers may be subject to airport security. In these cases, the container housing the samples may be opened and the samples inspected and reinserted in an order not consistent with the original packaging. In these cases, it is imperative that each individual sample container have a permanent identifier either in indelible ink or as a label affixed on the side of the sample container (see Section 10.2.4, “Container Label and Sample Identification Code”). Within each shipping container, a separate sample packing slip or tracking documents that lists the samples (by sample ID) for the container should be included.

Packages should be accepted only at designated receiving areas. Packages brought to any other location by a carrier should be redirected to the appropriate receiving area. All packages labeled RADIOACTIVE I, II, or III require immediate notification of the appropriate authorized user (NRC, 1998b).

A sample packing slip or tracking documents is required and must be presented at the time of receipt, and the approximate activity of the shipment should be compared to a list of acceptable quantities. If known, the activity of each radionuclide contained in the shipment must be

reviewed relative to the total amount of that radionuclide currently on site to ensure that the additional activity will not exceed that authorized by the NRC or Agreement State in the laboratory's license.

Surveying measures described in Section 11.3.2 may indicate that the samples are more radioactive than expected and that the radiation license limit may be exceeded. The laboratory should take extra precautions with these samples, but the survey results should be verified. The federal, state, or local agency should be contacted immediately when verified license limits are exceeded. The laboratory must respond quickly to stay in compliance with its license.

If the package is not accepted by the laboratory, the laboratory should follow corrective-action procedures prescribed in the radiation materials license, the appropriate plan document (if this is a reasonable possibility for the project), and the laboratory's SOPs. The project manager should be contacted about possible disposition of any samples.

11.3.2 Radiological Surveying

In addition to ensuring compliance with the laboratory's license and verifying estimates of radionuclide activity (Section 11.3.1), the radiological surveying of packages during sample receipt serves to identify and prevent the spread of external contamination. All packages containing samples for analysis received by the laboratory should be surveyed for external contamination using a wipe (sometimes referred to as a "swipe") and for surface exposure rate using the appropriate radiation survey meter. Exceptions may include known materials intended for analysis as: well-characterized samples, bioassays, or radon and associated decay products in charcoal media (exceptions should be listed in the laboratory SOP). Surveying of packages and sample containers received in the laboratory should be conducted in accordance with the laboratory's established, documented procedures and the laboratory radiation protection and health and safety plan. The exterior of the package is surveyed first; if there is no evidence of contamination or that the laboratory licence would be exceeded, the package is opened up and the sample containers surveyed individually. These procedures should include the action level and appropriate action as established by the facility. Personnel performing surveying procedures should be proficient in the use of portable radiation surveying instruments and knowledgeable in radiological contamination control procedures. Health and safety considerations are affected by the suspected or known concentrations of radionuclides in a sample or the total activity of a sample.

Radiation surveying is normally conducted using Geiger-Mueller (GM) detectors, ionization chambers, micro-R meters, or alpha scintillation probes, as appropriate. The laboratory should refer to any information they obtained before receipt of samples or with the samples, especially concerning the identity and concentration of radioactive and chemical constituents in the samples. Radiological surveying needs to be performed as soon as practical after receipt of the package, but not later than three hours (10 CFR 20.1906) after the package is received at the licensee's facility for packages received during normal working hours. For packages received

outside of normal working hours, the surveying must be performed no later than three hours from the beginning of the next workday.

Survey the exterior of a labeled package for radioactive contamination (10 CFR 20.1906). If the package is small (less than 100 cm²), the whole package should be wiped (swiped). Wipes are not always used, but if there is reason to believe that something has leaked, then wipes should be used. This survey is performed to detect possible violations of Department of Transportation (DOT) packaging and labeling regulations, as well as to determine the possible presence of gamma- and some beta-emitting radionuclides that may require special handling. Also, such a survey can help to avoid introducing a high-activity sample into a low-activity area. NRC (1998b) gives the following sample model for opening packages containing radioactive material:

- Wear gloves to prevent hand contamination.
- Visually inspect the package for any sign of damage (e.g. crushed, punctured). If damage is noted, stop and notify the RSO.
- Check DOT White I, Yellow II, or Yellow III label or packing slip for activity of contents, so shipment does not exceed license possession limits.
- Monitor the external surfaces of a labeled package according to specifications in Table 8.4, Section 13.14, Item 10 [of NRC, 1998b].
- Open the outer package (following supplier's directions if provided) and remove packing slip. Open inner package to verify contents (compare requisition, packing slip and label on the bottle or other container). Check integrity of the final source container (e.g., inspecting for breakage of seals or vials, loss of liquid, discoloration of packaging material, high count rate on smear). Again check that the shipment does not exceed license possession limits. If you find anything other than expected, stop and notify the RSO.
- Survey the packing material and packages for contamination before discarding. If contamination is found, treat them as radioactive waste. If no contamination is found, obliterate the radiation labels prior to discarding in the regular trash.
- Maintain records of receipt, package survey, and wipe test results.
- Notify the final carrier and by telephone, telegram, mailgram, or facsimile, the administrator of the appropriate NRC Regional Office listed in 10 CFR 20, Appendix D when removable radioactive surface contamination exceeds the limits of 10 CFR 71.87(i); or external radiation levels exceed the limits of 10 CFR 71.47.

In addition to these, laboratories may have additional internal notifications or procedures.

11.3.3 Corrective Action

The laboratory's SOPs should specify corrective actions for routine and non-routine sample problems, including deficiency in sample volume, leaking samples, and labeling errors. The appropriate corrective action may require consulting the project manager and other laboratory personnel. Timely response can allow for a broader range of options and minimize the impact of the sample problem on the project. The laboratory should document the problem, the cause (if known), the corrective action taken, and the resolution of each problem that requires corrective action. The documentation should be included in the project files.

11.4 Sample Inspection

After sample receipt, the next steps are to confirm that the correct sample has been sent, to check that the appropriate field preservation and processing have been performed, and to identify any hazardous chemicals.

Documents accompanying the samples should be reviewed upon receipt of the samples at the laboratory. If the proper paperwork is not present, the project manager should be notified. Data recorded on the paperwork, such as collection dates, sample descriptions, requested analyses, and field staff personnel, should be compared to data on the sample containers and other documentation. Any deficiencies or discrepancies should be recorded by the laboratory and reported to the project manager. The documents can provide data useful for health and safety surveying, tracking, and handling or processing of critical short-lived radionuclides.

11.4.1 Physical Integrity of Package and Sample Containers

Sample containers should be thoroughly inspected for evidence of sample leakage. Leakage can result from a loose lid, sample container puncture, or container breakage. Packages suspected to contain leaking sample containers should be placed in plastic bags. The authorized user or alternate authorized user must be notified immediately for assistance. If leakage has occurred, appropriate radiological and chemical contamination controls should be implemented. Sample materials that have leaked or spilled are normally not suitable for analysis and should be properly disposed. In all cases, the laboratory's management and project manager should be notified of leaks, breakage, spills, and the condition of sample materials that remain in the original containers.

Sample containers that have leaked (from a loose lid or puncture) may still hold enough sample for the requested analyses, so the laboratory should first determine whether sufficient representative sample remains. The sample is not usually analyzed if its integrity was compromised or is in doubt. Unless appropriate information is provided in the project plan or SOW, the project manager should determine whether or not the sample materials can be used for analysis or if new samples are required.

Packages, cooler chests, or individual sample containers may arrive at the laboratory bearing custody seals. These seals provide a means to detect unauthorized tampering. When packages or samples arrive with custody seals, they should be closely inspected for evidence of tampering. Custody seals are made from material that cannot be removed without tearing. If a custody seal is torn or absent, sample tampering may have occurred. This evidence of possible tampering is generally sufficient to preclude use of the sample for laboratory analyses. The project manager should be notified of the condition of the custody seal to determine if new samples are needed. Observations regarding the condition of the custody seals should be recorded according to the laboratory's standard procedures.

11.4.2 Sample Identity Confirmation

Visual inspection is the means to confirm that the correct sample has been received. Verifying the identity of a sample is a simple process where the appearance, sample container label, and chain-of-custody record or tracking documents are compared. If all three sources of information identify the same sample, then the sample is ready for the next step. If the sample label indicates the sample is a liquid and the container is full of soil, this discrepancy would indicate nonconformance. If the sample label states that there is 1,000 mL of liquid and there only appears to be 200 mL in the container, there may be nonconformance. Visual inspection can be used to:

- Verify identity of samples by matching container label IDs and tracking documents;
- Verify that the samples are as described by matrix and quantity;
- Check the tamper seal (if used);
- Verify field preparation (e.g., filtering, removing extraneous material), if indicated; and
- Note any changes to samples' physical characteristics that are different than those in the tracking documents.

11.4.3 Confirmation of Field Preservation

For those liquid samples requiring acid preservation, pH measurements may be performed on all or selected representative liquid samples to determine if acid has been added. The temperature of the sample may also be part of field preservation and the actual measured temperature should be compared to the specified requirements in the documentation.

11.4.4 Presence of Hazardous Materials

The presence of hazardous materials in a sample typically creates the need for additional health and safety precautions when handling, preparing, analyzing, and disposing samples. If there is

documentation on the presence of non-radiological hazardous constituents, the project manager should notify the laboratory about the presence of these chemicals. These chemical contaminants should be evaluated by the laboratory to determine the need for special precautions. The laboratory can also perform preliminary sample surveying for chemical contaminants using surveying devices such as a photoionization detector for volatile components. The presence of suspected or known hazardous materials in a sample should be identified, if possible, during project planning and documented in the plan document and SOW. Visual inspection can also be used such as checking the color of the sample (e.g., a green-colored water sample may indicate the presence of high chromium levels). The presence of suspected or known hazardous materials determined in the field should be communicated to the laboratory prior to the arrival of samples and noted on documentation accompanying the samples to the laboratory. If no documentation on non-radiological hazardous constituents is available, the laboratory should review previous experience concerning samples from the site to assess the likelihood of receiving samples with chemical contaminants. The laboratory's chemical hygiene officer and the project manager should be notified about the presence of potentially hazardous chemical contaminants.

11.4.5 Corrective Action

Visual inspection can also verify whether field sample preparation was performed as stated in accompanying documentation. Samples that were not filtered in the field or that reacted with the preservative to form a precipitate may represent a significant problem to the laboratory. If it appears that the sample was filtered in the field (e.g., there is no corresponding filter or there are obviously solid particles in a liquid sample), the liquid generally will be analyzed as originally specified. Laboratory personnel should check the project plan or SOW to see if the filter and filtered materials require analyses along with the filtered sample. If it appears that the sample was not filtered in the field (i.e., there is no corresponding filter or there are obviously solid particles in a liquid sample), sample documentation should be reviewed to determine if a deviation from the project plan was documented for the sample. It may be appropriate to filter the sample in the laboratory. The project manager should be notified immediately to discuss possible options such as filtering the sample at the laboratory or collecting additional samples.

One example of a corrective action for inspection is, if the pH is out of conformance, it may be possible to obtain a new sample. If it is not possible or practical to obtain a new sample, it may be possible to acidify the sample in the laboratory.

Visual inspection can serve to check certain aspects of sample collection. For example, if the SOP states that a soil sample is supposed to have twigs, grass, leaves, and stones larger than a certain size removed during sample collection and some of this foreign material is still included as part of the sample, this discrepancy results in a nonconformance.

11.5 Laboratory Sample Tracking

Sample tracking should be done to ensure that analytical results are reported for the “correct” sample. Sample tracking is a process by which the location and status of a sample can be identified and documented. The laboratory is responsible for sample tracking starting with receipt (at which time a unique laboratory sample ID is assigned), during sample preparation, and after the performance of analytical procedures until final sample disposition. The process of sample tracking begins the moment a field worker assigns an identification number (based on the information provided in the appropriate plan document) and documents how materials are collected. The way samples are transported from the field to the laboratory should be documented. The sample receipt procedures and documentation should be consistent when applicable with 10 CFR Part 20 Subpart J, and the client’s requirements as stated in the appropriate plan document or statement of work.

11.5.1 Sample Log-In

Laboratory sample IDs should be assigned to each sample in accordance with the laboratory’s SOP on sample codes. Each sample should receive a unique sample ID by which it can be logged into the LIMS, scheduled for analysis, tracked, and disposed. Information to be recorded during sample log-in should include the field sample identification number, laboratory sample ID, date and time samples were collected and received, reference date for decay calculations, method of shipment, shipping numbers, condition of samples, requested analyses, number and type of each sample, quality control requirements, special instructions, and other information relevant to the analysis (e.g., analytical requirements or MQOs) and tracking of samples at the laboratory. Laboratory sample tracking is a continuation of field sample tracking. Some of this information may have been entered into the LIMS during the planning phase.

Documents generated for laboratory sample tracking must be sufficient to verify the sample identity, that the sample may be reliably located, and that the right sample is analyzed for the right analyte. The documentation should include sample log-in records, the analysis request form, names of staff responsible for the work, when procedures are completed, and details concerning sample disposal. The documentation must conform to the laboratory’s SOPs.

During sample log-in, laboratory quality control (QC) samples may be scheduled for the analyses requested. The type and frequency of QC samples should be provided by the plan document or SOW and consistent with the laboratory’s SOPs.

11.5.2 Sample Tracking During Analyses

At this point, samples are introduced into the laboratory’s analytical processing system. The information gathered during surveying, along with the assigned tracking identification, passes to

the laboratory where specific preparation and analyses are performed. The sample may be further subsampled. Each subsample, along with the original sample, requires tracking to account for all materials handled and processed in the laboratory.

Each set of samples received by the laboratory should be accompanied by documents identifying the analytes required for each sample. These documents should be reviewed against the project plan documents or the SOW, which should identify the analytes, matrices, and analytical requirements and be part of the project documentation prior to the samples being received by the laboratory. Laboratory management personnel should be notified of any discrepancies. The requested analyses should be entered into the laboratory's tracking system. Typically, only one sample container of sufficient volume or quantity will be provided for a single or multiple set of different analyses. Each aliquant removed from the original container may require tracking (and perhaps a different laboratory sample ID).

Aliquants used during the analytical process can be tracked using analysis laboratory notebooks, forms, or bench sheets that record laboratory sample IDs, analyte, reference date for decay correction, aliquant size, and designated quality control samples. Bench sheets are loose-leaf or bound pages used to record information during laboratory work and are used to assist in sample tracking. Each sheet is helpful for identifying and processing samples in batches that include designated QC samples. The bench sheet, along with the laboratory log book, can later be used to record analytical information for use during the data review process. Bench sheets can also be used to indicate that sample aliquants were in the custody of authorized personnel during the analytical process.

After receipt, verification of sample information and requested analyses, and assignment of laboratory sample IDs, the requested analyses can be scheduled for performance in accordance with laboratory procedures. Using this system, the laboratory can formulate a work schedule, and completion dates can be projected.

11.5.3 Storage of Samples

If samples are to be stored and analyzed at a later date, they should be placed in a secure area. Before storage, any special preservation requirements, such as refrigeration or additives, should be determined.

The laboratory should keep records of the sample identities and the location of the sample containers. Unused sample aliquants should be returned to the storage area for final disposition. In addition, for some samples, depending on the level of radioactivity or hazardous constituents present, the laboratory should record when the sample was disposed and the location of the disposal facility. These records are necessary to ensure compliance with the laboratory's license for radioactive materials and other environmental regulations.

Areas where samples are stored should be designated and posted as radioactive materials storage areas. Depending on the activity level of the samples, storage areas may require special posting. If additional storage space or shielding is needed, arrangements that are consistent with the license should be made with the authorized user. See Chapter 17 for more information on waste disposal.

11.6 References

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