

Hanford Analytical Services Quality Assurance Requirements Document

Volume 2: Sampling Technical Requirements

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management



ENERGY | Richland Operations
Office

P.O. Box 550
Richland, Washington 99352

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LIST OF TERMS

AEA	Alpha Energy Analysis
ALARA	As Low As Reasonably Achievable
ASTM	American Society for Testing and Materials
CFR	Code of Federal Regulations
DOE	U.S. Department of Energy
DOT	U.S. Department of Transportation
DQO	Data Quality Objective
EMS	Environmental Management System
EPA	U.S. Environmental Protection Agency
GEA	Gamma Energy Analysis
HASQARD	Hanford Analytical Services Quality Assurance Requirements Document
IATA	International Air Transport Association
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ISMS	Integrated Safety Management System
PCB	Polychlorinated Biphenyls
QA	Quality Assurance
QC	Quality Control
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SVOA	Semivolatile Organics Analysis
TCLP	Toxicity Characteristic Leaching Procedure
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WAC	Washington Administrative Code

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1.0 SAMPLING AND ANALYSIS PROCESS

1.1 INTRODUCTION

The *Hanford Analytical Services Quality Assurance Requirements Document* (HASQARD) is made up of four volumes: Volume 1, *Administrative Requirements*; Volume 2, *Sampling Technical Requirements*; Volume 3, *Field Analytical Technical Requirements*; and Volume 4, *Laboratory Technical Requirements*. Volume 1 describes the administrative requirements applicable to each of the other three volumes and is intended to be used in conjunction with the other three technical volumes. As noted in Volume 1, Section 1.1, the HASQARD identifies the quality principles, practices, and processes for sampling and for the analytical service provider's quality assurance (QA) documents covering regulatory analysis (e.g., Tri-Party Agreement, permits, process chemistry, and research and development efforts related to the Hanford Site clean-up activities). Volumes 1 and 2 together describe the administrative and technical requirements for sample collection and handling.

1.2 SCOPE

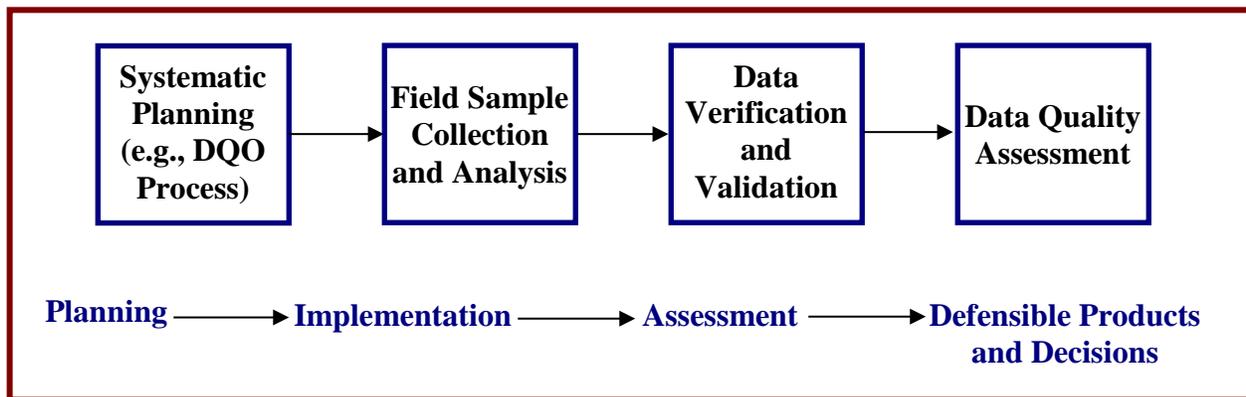
Volume 2 addresses the technical requirements related to sample collection and handling. Section 2.0 briefly indicates the necessity and importance of proper planning in delineating technical requirements, and indicates consensus standards and regulatory guidance for technical sampling requirements. Section 3.0 discusses the role of the Integrated Safety Management System/Environmental Management System (ISMS/EMS) in sampling operations. Section 4.0 on Sampling Operations addresses technical requirements related to documentation of sampling activities, sample identification, sample preservation, sample storage, sample handling and transfer, sample packaging and shipping, sample custody, sample holding times, sample containers, and field change requirements. Section 5.0 on Quality Assurance and Quality Control primarily addresses review of field documents and technical requirements related to different types of field quality control (QC) samples such as blanks, replicates, and spikes.

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2.0 THE PLANNING PROCESS IN SAMPLE COLLECTION

The project-level life cycle quality processes (adapted from EPA QA/G-4, EPA/240/B-06/001 February 2006, *Guidance on Systematic Planning Using the Data Quality Objectives Process*) depicted in Figure 2-1 center around the planning, collection, and evaluation of analytical and sampling data.

Figure 2-1. Project-Level Life Cycle Quality Components.



Planning for environmental activities involves a systematic approach to ensure that data or information is of the needed and expected quality for the desired use. There are different variations to systematic planning, tailored to specific application areas. For example, the Observational Method is a variation on systematic planning that is used by many engineering professions. The Triad Approach, developed by the U.S. Environmental Protection Agency's (EPA's) Technology Innovation Program, combines systematic planning with more recent technology advancements, such as techniques that allow for results of early sampling to inform the direction of future sampling. However, it is the Data Quality Objective (DQO) Process that is the most commonly-used application of systematic planning in the general environmental community and is the most commonly used application of systematic planning at the Hanford Site.

The DQO process is a logical method that clarifies the problems or questions that a program must solve. It further defines the information needed to answer questions and objectives, including an assessment of risk or uncertainty. The DQO process may be applied to either programmatic or technical issues.

The planning process (DQO process) is also a critical element relative to QC. The DQO process identifies the quality needed by the analytical data in order for the data to be useful for their intended purpose. Further, the DQO may specify QC acceptance and performance requirements to be met by the data (i.e., requirements related to accuracy, precision, completeness, reproducibility, and comparability).

Because the DQO process is described in Section 1.3 of Volume 1 and more extensively in Appendix B of Volume 1, it will not be further described in this volume of the HASQARD.

2.1 IMPLEMENTATION OF PLANNING PROCESS

NOTE: *The terminology Sampling and Analysis Plan (SAP) is used throughout this document because of its common usage in Comprehensive Environmental Response, Compensation, and Liability Act driven characterization processes. However, SAP shall be understood to be a generic term in this volume of the HASQARD, and different Hanford Site organizations may use different terminology to refer to the document(s) which implement the DQO. The generic usage of SAP encompasses any formal document which specifies sampling and analysis requirements including project-specific work packages, sampling instructions, project-specific QA documents, field sampling plans, etc. for specific sampling events.*

The SAP implements the DQO by making clear the sample and analytical data QC requirements. The SAP specifies QC requirements for field sampling and measurement processes, requirements for laboratory-generated analytical data, and requirements for traceability and defensibility, as well as for quality processes such as data verification and data validation after data has been received from the laboratory. The design of a field sampling effort should be performed as a part of the DQO process, and details should be incorporated into the SAP.

Each prime contractor at the Hanford Site shall have a QA plan and/or procedure(s) which address the use and documentation of DQOs. Further, each prime contractor at the Hanford Site shall have a QA plan and/or a standard operating procedure (SOP) which addresses the use, content, and format of SAPs. Each commonly used sampling method performed in the field shall have an applicable SOP describing the necessary equipment and collection steps for the media and contaminant to be sampled. Unique sampling methods may be described in the SAP document(s) to accommodate unusual field conditions or limitations imposed due to safety considerations or to technical data needs.

Project management, in conjunction with personnel knowledgeable in the relevant analytical criteria, shall develop, establish, and update sampling and analytical data deliverable requirements based on project DQOs. Each project or program shall identify and clearly define specific data deliverables expected from the sampling organization supporting its work. These deliverables shall be designed to ensure project information contains the appropriate QC and documentation.

Approved and maintained documents shall be in place to address data deliverable requirements that meet project requirements. Sampling organizations providing projects with samples and data shall be aware of deliverable requirements and be able to provide the stated deliverables in a consistent and timely manner. If project management determines that existing formally approved documents are sufficient to meet or exceed project needs relative to data deliverable requirements, new documents need not be developed.

If data deliverables include electronic files, reporting formats shall be compatible with the project's system. Standard formats for transmission and database structure requirements shall

include consistency with the Hanford Site standards (e.g., Hanford Environmental Information System, Tank Waste Information Network System, Format for Electronic Analytical Data) for collecting, storing, transmitting, and evaluating environmental data.

2.1.1 Limitations and Restrictions for Highly-Radioactive Samples

Highly-radioactive samples such as those likely to be encountered in tank wastes, in soils underlying cribs, and in waste pits pose unique sampling and analysis problems. Highly-radioactive samples may have sampling/handling activities, sample volumes, containers, or preservation restricted by the limitations (e.g., sample retrieval methodology, equipment, or by shielded containers required for sample storage and transportation) necessary to address safety related concerns associated with the As Low As Reasonable Achievable (ALARA) principle. Highly-radioactive samples typically require that different collection and handling procedures be used than for more low-level environmental samples. Highly-radioactive samples generally will be taken with minimum handling.

Requirements and guidance in the sections which follow may not be relevant for highly-radioactive samples. Consequently, pre-sampling activities are particularly important when highly-radioactive samples may be encountered. The consequences and impacts of the limitations imposed by high activity in the sample on data quality shall be taken into account in the sample collection planning process (Section 2.0). Sections in project-specific SOPs, SAPs, or work control documentation shall address how highly-radioactive samples will be collected, preserved, handled, packaged, and shipped.

2.2 REGULATORY, CONSENSUS, AND GUIDANCE SAMPLING STANDARDS

Documentation of sampling procedures is critical to the technical defensibility and the legal defensibility/admissibility of the resulting data. Whenever possible, industry-recognized sampling methods from agency-published source documents, such as U.S. Department of Energy (DOE), EPA, and American Society for Testing and Materials (ASTM), should be employed. Sample collection and processing procedures may also use methods published by the U.S. Geological Survey, U.S. Department of Agriculture, and professional groups such as the American Water Works Association. Current reference sampling methods detailed in the following sources include, but are not limited to:

- EPA 540-R-09-03, *Contract Laboratory Program Guidance for Field Samplers*, OSWER 9240.0-47, January 2011.
- EPA 402-R-97-016, Rev. 1, *Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)*, August 2000.
- SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, 3rd Edition as amended.
- NAVSEA T0300-AZ-PRO-010, Rev. 1, *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*, August 2009.

- EPA 530-D-02-002, *RCRA Waste Sampling Draft Technical Guidance*, August 2002.
- ASTM D5730-04, *Standard Guide for Site Characterization for Environmental Purposes with Emphasis on Soil, Rock, the Vadose Zone and Ground Water*.
- EPA/240/B-06/001, *Guidance on Systematic Planning Using the Data Quality Objectives Process*, EPA QA/G-4, February 2006.
- EPA/240/R-02/005, *Guidance on Choosing a Sampling Design for Environmental Data Collection*, EPA QA/G-5S, December 2002.
- Washington State Department of Ecology Toxics Cleanup Program, *Guidance on Sampling and Data Analysis Methods*, Publication No. 94-49, January 1995.
- HASL-300, *EML Procedures Manual*.
- PNNL-19915, *Visual Sample Plan (current version) User's Guide*.

Sampling methods employed that are not found in agency-published source documents should be thoroughly reviewed and approved by the cognizant project management and/or QA organization prior to implementation. If specific method references do not exist, appropriate documents, such as suppliers' manuals, equipment manufacturer instructions, and instrumentation specifications are recommended for use provided such documents include adequate descriptions and criteria to ensure the required quality of work performed.

3.0 SAMPLING SYSTEM GUIDING PRINCIPLES

Sampling operations line management shall develop, establish, and update requirements for sampling organizations and personnel qualifications, personnel training, site guidelines, sampling methods, procedures, corrective actions, document control, and field assessments. Approved and maintained procedures shall be in place. Program/project management identifies specifications and ensures they are satisfied.

Defining the scope of work, identifying hazards, developing and implementing hazard controls, and working within controls are key elements of ISMS (DEAR 952.223-71, *Integration of Environment, Safety, and Health into Work Planning and Execution*) and EMS (DOE O 436.1, *Departmental Sustainability*) programs. To address those key elements the following conditions shall be considered by sampling management and personnel. The list below should be considered representative of key issues and not all-inclusive of all ISMS/EMS principles potentially applicable to sampling activities.

- Sampling operations should be planned prior to implementation in the field.
- Consideration shall be given as to whether Hanford Site facilities are secure and as to whether buildings, field laboratories, and controlled sampling points (e.g., monitoring wells) have access limited to authorized personnel.
- Adequate storage areas for reagents, solvents, standards, glassware, containers, samples during interim storage, and reference materials shall be established to prevent cross contamination or degradation.
- Instrumentation, equipment, and utilities shall be maintained to perform the required/contracted sampling operation. Safety equipment will be available and readily accessible. Sampling equipment shall be kept secured when not in use.
- Surface disturbances such as pits, holes, excavations, and trenches will be clearly marked or barricaded. Addition of new surface features (e.g., well heads, pumps, piping, and electrical traces) will also be clearly marked.
- Sampling designs shall minimize interactions between high and low concentration areas and shall minimize common utilization of equipment, instrumentation, and facilities. A formal, active contamination control that minimizes the potential spread of contamination between sample processing and sample storage areas will meet the fundamental elements of an active ALARA program. Specially-controlled facilities or areas shall be established for the receipt of highly contaminated materials and storage of samples.
- Sampling designs shall, to the extent possible, ensure that samples to be collected will be representative of the material from which they are derived. Project-specific SOPs, SAPs, or work control documentation shall provide field sampling personnel with direction on the

collection of quality and representative samples relative to the sampled media and analytical test to be performed on the sample.

- Line management is responsible for ensuring that waste disposition and worker health and safety are adequately addressed.
- Design and implementation of sampling programs shall address situations or conditions necessary for the controlled use, storage, and disposition of sample material rejects (e.g., soil discards, purged waters), equipment decontamination residues, and/or remnants of samples. These programs also ensure that all activities that may impact environmental data are documented and recorded in a field logbook (see Washington Administrative Code (WAC) 173-303-210, “Generator Recordkeeping,” and WAC 173-303-380, “Facility Recordkeeping,” for more information).

4.0 SAMPLING OPERATIONS

Project management, in conjunction with personnel knowledgeable in the relevant sampling criteria, shall develop, establish, and update requirements for management of documentation, sample collection, waste disposition, chain-of-custody, sub-sampling, holding times, and sample containers. If project management determines that existing sampling SOPs or other approved work control documents are sufficient to meet or exceed project needs, new documents need not be developed.

4.1 PRE-SAMPLING ACTIVITIES

Generally, a variety of activities and tasks take place before field sampling can occur. Such activities may include, but are not limited to, kick-off meetings, coordination meetings, pre-job briefings, site walk-downs, site surveys, generation of maps designating sampling locations, notifications to analytical laboratories, generation of various paperwork for samplers and for analytical laboratories, generation of labels and chain-of-custody forms, and reviews of various paperwork and documents, ensuring sufficient sample bottles and other supplies are available, and field sampling crew planning as applicable. Project-specific SOPs, SAPs, or work control documentation shall describe pre-sampling activities as appropriate.

4.2 SITE/FIELD DOCUMENTATION

Site and field documentation includes work control documents, logbooks, and data forms. Work control documents provide the field sampling implementation details needed to successfully conduct a sampling event. Logbooks and data forms are a means for providing a daily record of all field activities at an investigation site and are generally considered the primary record for sampling activities. QA requirements in project-specific SOPs, SAPs, or work control documentation determine the type and extent of documentation needed at a site.

4.2.1 Work Control Documents

The scope of work performed for a specific sampling activity includes work control documents detailing the work to be performed in the field. Work control documents may include work packages and technical procedures used to perform the sampling activity. Work control documents are determined during the work planning process where they are planned, reviewed, and approved prior to performing the sampling activity. Work control document files/records may contain but are not limited to the following:

- Radiological Work Permits
- Environmental Compliance Forms
- Technical Safety Requirements
- Chain-of-Custody Records
- Waste Planning Checklists
- Pre-Job Briefings
- Temporary Shielding Authorization Forms

- Job Hazard Analysis Checklists
- Industrial Hygiene Sample Plan
- Sampling Procedures
- Sample Data Sheets
- Sampling Analysis Plan
- Glove Bag Certification Checklists
- Prerequisite, Daily and Work Complete Checklists.

4.2.2 Logbooks

Logbooks provide a convenient tool for documenting the details of field activities performed in support of sampling events. Entries to a logbook shall be made on a real-time basis when possible and in chronological order. The logbook entry shall be completed and signed daily or as soon as practicable upon conclusion of the recorded activities by the person responsible for completing the recorded activities. If activities carry over to additional logbook pages, each completed page will be signed and dated by the individual responsible for the activity that carries over to the next page.

All logbooks shall be bound. Logbooks shall have ruled and sequentially numbered pages. All logbook entries will be made in indelible ink and be made in a manner such that they can be easily read, understood, and reproduced with a standard photocopier. Write-overs are not permitted. Corrections are made by marking the erroneous data through with a single line, entering the correct data, and initialing and dating the changes. Correction fluid and erasers are not to be used. Pages will not be removed from logbooks for any reason prior to final records retirement of the logbook. Empty spaces shall be lined through and initialed and dated at the completion of an entry to prevent non-related or unauthorized entries from being added to the logbook. Blank pages between logbook entries shall be marked "page intentionally left blank" or shall be lined through, initialed, and dated. Only authorized persons may make entries in logbooks.

Any attachments taped into the logbook must be taped completely around the attachment. Attachments must be dated and initialed by the person making the logbook entry such that the date and initials extend from the attachment, across the tape, and onto the logbook page. Attachments relating to a particular sampling event may be added to the logbook after the fact (e.g., as materials become available). In such occurrences, a reference to the original sampling event will be made (date or page number) or the "continued from" (or "continued on") section of the logbook page employed.

Electronic logbooks are permitted and shall meet the same requirements for change protection and control as hand-written hardcopy logbooks.

4.2.2.1 Site Logbooks

In certain circumstances, sites or facilities to be sampled may have a site logbook. These are generally large sites which are entered repeatedly over long-time durations. A site logbook is the

master reference document for activities performed at such sites. A site logbook is not necessary at every site to be sampled, and the use of a site logbook should be specified in a project execution plan or similar document. The decision to use a site logbook and maintenance of the site logbook is the responsibility of the specific project accountable for the site, not the sampling organization.

Entries to a site logbook shall be made and initialed on a real-time basis, with summaries completed at the end of each sampling event when the site is exited. The site logbook must be identified with a unique project name and number on the front or inside cover. Start and completion dates shall be indicated by documentation within the logbook. Any onsite incidents and any injuries shall be recorded in the site logbook. The site manager, site supervisor, or other designated project personnel shall review entries for completeness and document the review with signature and date.

The following items shall be recorded in the site logbook as applicable:

- The day, date, time arrived onsite, names, titles, organizations of personnel present onsite.
- The purpose of the visit.
- All site activities, including field tests. The site logbook summarizes daily activities and should therefore not be as detailed as field logbooks.
- Any equipment failures or breakdowns, with a brief description of repairs or replacements made, and indications of the impact of the equipment failure.

4.2.2.2 Field Logbooks

Field logbooks contain area- or task-specific information. The field logbook cover shall indicate the particular tasks, or areas, or the specific individuals to which the logbook is assigned. The field logbook shall be identified as a record and shall be maintained as such. The field logbook is the responsibility of the organization performing the field activity. Field logbooks shall be water resistant.

Information to be recorded in the field logbook (or data forms, Section 4.2.3), as appropriate, includes the items below. Information recorded on data forms, per Section 4.2.3, does not need to be duplicated in the field logbook unless specifically requested.

Results of any field activity including field calibrations and surveys shall be noted and annotated in the field logbook. Other details and information needed to fully describe the field activity shall otherwise be noted and appear in the field logbook. The sampler or task leader shall review and sign the field logbook. The field manager, supervisor, or cognizant scientist/engineer shall review entries and document the review with signature and date.

The following items shall be recorded in the field logbook as applicable to the field activities being performed:

- The day and date, time the task started, weather conditions, and the names, titles, and organizations of personnel performing the task.
- The purpose of the visit to the task area.
- Site activities in specific detail (e.g., maps and drawings) or the forms used to record such information (e.g., soil boring log or well completion log). Details of any field tests that were conducted. Reference any forms that were used, other data records, and the SOPs followed in conducting the activity.
- Details of any field calibrations and surveys that were conducted. Reference any forms that were used, other data records, and the SOPs followed in conducting the calibrations and surveys.
- Details of any samples collected and indicate the preparation, if any, of splits, duplicates, matrix spikes, or blanks. Reference the SOPs followed in sample collection or preparation. List location of sample collected, sample type, all label or tag numbers, sample identification, sample containers and volume, preservation method, packaging, chain-of-custody form number, and the analytical request form number pertinent to each sample or sample set. Note the time and the name of the individual to whom custody of samples was transferred.
- The time, equipment type, and serial or identification number, and the SOP followed for decontaminations and equipment maintenance carried out. Reference the page number(s) of any logbook (if any) where detailed information is recorded.
- Any equipment failures or breakdowns that occurred, with a brief description of repairs or replacements.

4.2.3 Data Forms

It is often convenient to document field information on pre-printed data forms. As with logbooks, data forms shall be completed with indelible ink and may be considered a record. If the completed data form is deemed to be a record it shall be maintained and stored as such. Write-overs are not permitted. Corrections are made by marking the erroneous data through with a single line, entering the correct data, and initialing and dating the changes. Correction fluid and erasers are not to be used. Unused data form fields or spaces shall be lined-out with initials and date, or marked "N/A."

Alternatively, as noted in Section 4.2.4, data forms may be completed electronically.

4.2.4 Field Electronic Data Gathering

Automated data entry systems for field data collection may be used. Electronic notebooks and data loggers can reduce repetitive data entry and transcription errors. Field computers may be used to input field information and data on electronic data forms, to add information and complete electronic chain-of-custody forms, and to add information as required in Sections 4.2.1 and 4.2.2 to electronic notebooks. Control of software used for field electronic data gathering shall be implemented per HASQARD Volume 1, "Software Systems Quality Assurance," requirements.

4.3 SAMPLE COLLECTION

When performing a sampling event, samplers are expected to follow SOPs or prescribed sampling techniques. Matrices include, but are not limited to, soil, water, sediment, or various waste types. The requirements for collecting each matrix type should be specified in the project-specific SOPs, SAP, or other work control documents.

4.3.1 Order of Sample Collection

When multiple sample bottles are filled during a single sampling event, the order of collection should be based upon the potential loss of volatile components or degradation of unstable components during sampling. Samples are recommended to be collected in the following order or as designated in the project-specific SOPs, SAP, or other work control documents:

- a. Water quality sample (if applicable) for determination of field measurements
- b. Volatile organic compounds
- c. Total organic halogens
- d. Total organic carbon
- e. Extractable organic compounds (semi-volatiles, pesticides, polychlorinated biphenyls (PCBs))
- f. Remaining inorganic and radiological analytes.

NOTE: *Because tritium (H^3) is associated with soil moisture and surface soils at the Hanford Site typically desiccate rapidly, soil samples for tritium analysis must be collected from ~0.3 m (~1.0 ft) below ground surface. Sample containers should be filled as full as practicable with soil and the container lids should be tightened quickly to avoid soil moisture evaporation.*

Project-specific requirements for container filling may supplant or modify this sample collection order. Project-specific container filling order should be specified in the SOPs, SAP, or other work control documents.

4.3.2 Sub-Sampling, Compositing, and Homogenizing

Samples collected in the field may have to be composited, or homogenized, or sub-sampled. How such activities are performed should be described in the project-specific SOPs, SAP, or other work control documents. If unexpected situations occur during sampling such that

instructions provided in these documents are not applicable, actual methods of compositing, homogenizing, or sub-sampling shall be described in detail in the field logbook.

4.3.3 Collection of Highly-Radioactive Samples

Highly-radioactive samples may have sample volumes restricted. Restricted sample sizes may have significant impacts on the amount of material available for analysis of the samples; particularly where multiple different types of analyses have to be performed on the sample. Limited sample sizes may have impacts on the types of laboratory QC samples run per batch and on detection limits.

4.4 MANAGEMENT OF SAMPLES

Samples may be collected from known or suspected hazardous sites that contain hazardous organic, inorganic, and/or radiochemical materials. Sampling organizations must be aware of potential hazards associated with the collection, handling, and disposition of these samples. The sampling team shall be provided with historical and background information on the potentially contaminated site to give them guidance on health and safety precautions that should be initiated. It is the responsibility of the sampling organization to take necessary measures to ensure the health and safety of its employees, to follow ALARA principles, and to meet regulatory requirements.

During the pre-job planning phase, the field organization should be cognizant of any special requirements that come into effect when working with listed waste, environmental media that contains listed waste, and hazardous debris containing a listed waste. Communication with the laboratory pertaining to these issues should occur prior to sample collection and delivery.

4.4.1 Sample Identification

The project-specific SOPs, SAP, or other work control documents shall describe methods to ensure that samples are identified and controlled in a consistent manner. The identification system shall ensure traceability of samples from time and place of collection through shipment to authorized persons or organizations and/or disposition. The identification of QC samples shall be contained within project documentation to allow the relationship of QC data to specific samples to be traceable.

Samples have their own unique identification numbers. The sample identification number is a critical link in the traceability of analytical data to the project. This number shall be recorded in the appropriate field and project documentation (i.e., chain-of-custody forms and/or field data sheets) with information describing the sample. Each sample is identified by affixing a standardized label or tag on the container. This label or tag shall contain the sample identification number. In addition, the label shall identify or provide reference to associate the sample with the date and time of collection, preservative used (if applicable), analysis required, and the collector's name or initials. Sample labels may be pre-printed or hand written. In either event, the ink should be indelible or waterproof. The label is placed on the outside of the sample

bottle or container, and if waterproof labels are not used, then the labels should be covered with clear packaging tape to protect the label and maintain legibility.

4.4.2 Sample Custody

A major consideration for the legal and technical credibility of analytical data generated from a field sampling activity and subsequent sample analysis is the ability to demonstrate that samples have been collected by the sampling group and have reached the laboratory without alteration. Evidence of collection, temporary storage, and shipment to the laboratory shall be documented.

Documentation is accomplished through chain-of-custody processes and records which describe and document: (1) how physical custody is maintained, (2) how custody is transferred, (3) the individuals responsible for sample collection, and (4) the method for sample processing, shipping, storing, and dispositioning. A sample is considered in custody if (1) it is in the physical possession of a custodian (refer to Section 4.4.6 for custodian responsibilities), (2) it is maintained in visual contact by a custodian so they can verify that it has not been tampered with, (3) it is locked up or within a sealed container so the custodian can verify that no one has tampered with it, or (4) it is stored in a restricted area and is accessible only to authorized personnel trained to procedures regarding maintaining sample integrity and custody control within the restricted area.

The field sampling organization will establish SOPs that describe the interface and custody responsibilities for sample collection, temporary storage, custody transfer, shipping of the samples to the final destination, and disposition.

The following minimum information is required on the completed chain-of-custody form:

- Collector(s) names
- Project designation
- Unique sample numbers
- Date, time, and location (or traceable reference thereto) of sample collection
- Chain of possession information (i.e., signatures and printed names of all individuals involved in the transfer of sample custody and storage locations, dates of receipt and relinquishment).

Additionally, the following information could be included on the chain-of-custody:

- Sample matrix
- Sample preservation used (if applicable)
- Shipped to (i.e., analytical laboratory performing the analysis)

- Requested sample analysis method (i.e., analysis to be performed by the analytical laboratory)
- Required turn-around time
- Logbook or work package identification number/page number used to document field sampling activity information
- Container type and number
- Special instruction for the analytical laboratory.

Chain-of-custody forms shall accompany samples delivered to the laboratory facility(ies) that is performing the analyses. These forms shall be signed and dated upon receipt in the facility. Where practicable, the laboratory shall verify that the inventory of samples in the delivered sample containers matches the samples listed on the chain-of-custody form(s) before signing and dating the chain-of-custody form(s). Otherwise, the laboratory shall sign and date the chain-of-custody form(s) in accordance with laboratory custody procedures.

To the extent practicable, chain-of-custody forms shall be protected to prevent tampering or damage. Chain-of-custody forms shall accompany samples from the point of sample collection through receipt at the laboratory.

When samples are relinquished to a shipping company for transport in a custody-sealed shipping container, the shipping company shall provide a shipping bill/receipt. Employees of the shipping firm do not sign the chain-of-custody. The tracking number from the shipping bill/receipt is to be recorded on the chain-of-custody form or in the project documentation. See Section 4.4.6, "Sample Handling and Transfer," for further information related to the transfer of samples.

Agreement shall be reached between the laboratory and customer regarding disposition of the original custody form (i.e., retained by the laboratory, returned immediately to the customer, delivered to the customer as part of the final data deliverable). Chain-of-custody forms are to be reviewed for accuracy.

4.4.3 Sample Containers

The project-specific SOPs, SAP, or other work control documents shall specify types of sample containers and the level of cleanliness required. The potential impacts of special or non-standard sample containers on analytical data quality should be addressed in the DQO.

Samples shall be collected, where and when appropriate, in break-resistant containers. The field sample collection record shall indicate the laboratory lot number of the bottles used in sample collection. When commercially pre-cleaned containers are used in the field, the name of the manufacturer, the lot identification, and certification shall be retained for documentation.

Containers shall be capped and stored in an environment which minimizes the possibility of contamination of the sample containers. If contamination of the stored sample containers occurs, corrective actions shall be implemented to prevent reoccurrences. Contaminated sample containers cannot be used for a sampling event. Contaminants in this case are defined to be semi-volatile chemicals such as spray lubricants, cleaning chemicals, etc. that if leaked, spilled, sprayed, used, or otherwise discharged have the potential to contaminate the sample containers.

4.4.3.1 Sample Containers for Highly-Radioactive Samples

Glass containers with Teflon-lined septums or caps are required for most organic analyses and zero headspace is required for volatile organic analysis (VOA). However, the high radiation levels may make remote or modified handling necessary. This may make zero headspace sampling impractical and nearly impossible. The headspace should be minimized as much as sampling constraints will allow.

4.4.4 Sample Preservation

Because of the time lag that occurs when samples are transported from the field to an analytical laboratory, some preservation is necessary to maintain the integrity of the samples. Samples shall be preserved in a manner consistent with regulatory requirements and with established analytical method requirements.

Sample preservatives should be added to the sample container prior to sample collection, or immediately upon sample collection whenever possible. In some instances, samples may be preserved by the laboratory upon receipt. Case-by-case preservation decisions shall be made based on laboratory requirements, matrix concerns, DQOs, and state or Federal regulations. U.S. Department of Transportation (DOT) regulations apply to pre-preserved containers, preservatives transported to the field, and preserved samples. Sample preservation and extension of holding times may be negotiated with the regulators to support cost-effective collection of data with known and controlled sources of variability. This analyte- and sample-specific approach is consistent with EPA processes for DQOs and data quality assessments.

Methods of preservation are relatively limited and generally are intended to: (1) retard biological action, (2) retard hydrolysis and radiolysis of chemical compounds and complexes, (3) reduce volatility of constituents, and (4) reduce absorption and adsorption effects. Preservation methods generally are limited to pH control, chemical addition, refrigeration, and freezing. No single standard method of preservation and storage can be recommended for samples. Generally, the analytical method procedure will specify the acceptable preservation technique. In addition, the project-specific SOPs, SAP, or other work control documents shall specify required preservation techniques.

The method of preservation shall be recorded in the field documentation along with other pertinent information required by the SOP. Preservatives shall be tracked by lot number, date of receipt, and date opened. Any use of chemical preservatives shall be indicated on the sample container label. If samples require preservation by freezing, then care shall be taken to ensure

sample containers are not overfilled with sample media that may expand and burst the sample container.

Appendix A contains a compilation of example general preservation guidelines for various types of analyses for both soil and water matrices. It is not intended to be comprehensive but is representative of most typical environmental sampling at Hanford. Project-specific SOPs, SAP, or other work control documents should specify preservation requirements for the specific sampling events described in these documents.

4.4.4.1 Preservation of Highly-Radioactive Samples

Many methods, including SW-846 methods, require samples to be cooled while awaiting analysis. Highly-radioactive samples may be transported and stored in shielded casks, which precludes sample cooling. In addition, some of the waste inherently generates heat and cannot be cooled to the required temperature without extreme measures being taken.

Samples of aqueous liquids typically require acidification to pH 2 for preservation before most metal analyses. Handling of the samples in the field for acid addition and pH verification may not be desirable from an ALARA standpoint. Also, samples (e.g., core samples) may be collected and shipped in special vessels that cannot be accessed to adjust the pH. In many cases, the presence of liquid materials will not be known until the sample is extruded from the sample core collection barrel. Preserving some liquid samples by adding acid could require a large amount of acid that would alter the chemical and physical characteristics of the waste. Some liquids (e.g., Hanford Site tank wastes) contain high levels of dissolved salt which could precipitate solids when preserved by adding acid. This would adversely affect the goal of assessing concentrations and physical properties of the sample.

4.4.5 Sample Storage

Site storage can be minimized by coordinating a sample shipment schedule with the laboratory. Storage areas shall be dedicated to samples only and controlled to prevent damage or loss, and to maintain sample container and identification integrity. Measures shall be taken to avoid sample contamination during storage. Measures also shall be taken to contain and avoid material spills during storage.

When storage is necessary, the samples shall be stored in predetermined physical and environmental conditions commensurate with the intended analysis and regulatory requirements specific for the analyte and matrix. Daily verification and documentation of storage temperature shall be maintained on each scheduled work day in accordance with the project-specific SOPs, SAP, or other work control documents. Storage blanks shall be used as appropriate.

4.4.6 Sample Handling and Transfer

One member of the sampling team shall be identified as the sample custodian (**NOTE:** *Different sampling organizations may use different names for the custodian, such as field work supervisor. Because different sampling organizations may use different names for the custodian position, the*

term “custodian” shall be understood to be a generic position descriptor). The custodian is responsible for maintaining custody of the samples and for maintaining the chain of custody while samples are in the custodian’s possession. The custodian, the collector (as identified on the chain-of-custody form), and the first person relinquishing possession of the samples shall be the same. The custodian documents each transaction, and the sample remains in the custodian's possession until relinquished. Each time the custody of samples is turned over to another person, the chain-of-custody form must be signed off by the former custodian and accepted by the new custodian.

SOPs shall establish methods to control samples during handling and transfer to preclude loss of identity, damage, deterioration, and loss of sample. Chain-of-custody documentation accompanying samples will be maintained at all times. The sample identification number shall be marked on the sample container and the chain-of-custody form.

The custodian is responsible for properly packaging and dispatching samples to the appropriate laboratory or facility. This responsibility includes completing, dating, and signing the appropriate portion of the chain-of-custody form, sample transfer, and shipping forms (as applicable). Verification of sample identification and integrity shall be performed prior to acceptance of the sample from another staff member or organization for field analysis, introduction into storage, or delivery to the designated laboratory. When transferring the samples, the person who accepts the samples shall legibly print and sign their name and record the date and time of the transfer on the chain-of-custody form. If the transfer of custody is between companies, the company affiliation along with the signatures must be noted.

Precautions shall be taken not to contaminate samples or field personnel. The outside of the container shall be wiped clean of any visible dirt, grime, or liquid after the sample has been placed in the container. When working in a radiological controlled area, the container shall be surveyed according to site-specific procedures. Precautions shall be taken to ensure that the outside of the container does not become contaminated (e.g., placing the container in a plastic bag or some other protection).

Custody seals or custody tape shall be used to verify that sample integrity has been maintained during transport. The cap of the individual sample container shall be sealed so that any tampering is easy to detect. When it is not practical to apply the custody seal or custody tape directly on the cap of the sample container, the sample container shall be placed inside a secondary container (e.g., larger sample container) that is sealed with a custody seal. This secondary container cannot also be the shipping container. Custody seals or tape shall also be applied to the sample shipping container prior to offsite transport or during temporary storage during work breaks to verify that sample integrity has been maintained. Custody seals or tape shall be selected that is not removable from the shipping container without breaking the seal. Samples shall be shipped in insulated containers with either synthetic ice or ice packed in plastic bags when samples require cooling. Overcooling of samples shall be avoided to ensure that sample integrity is not compromised.

The sample container(s) shall be placed in a transportation case. Pertinent field records, analysis request forms, and chain-of-custody form may be included in the transportation case or accompany the samples. A copy of each form shall be retained by the originating organization. The transportation case shall be secured, labeled, and marked in accordance with appropriate DOT/International Air Transport Association (IATA) regulations.

4.4.7 Sample Screening, Packaging and Shipping

Instructions for screening, packaging, and shipping of samples shall be established in a SOP. The transportation of samples shall be accomplished in a manner designed to protect the integrity of the sample(s) and in such a way that prevents any detrimental effects to personnel, the public, and the environment from potentially hazardous samples.

All packaging and transportation instructions shall be in compliance with applicable transportation regulations, DOE requirements, and contractor requirements. Regulations for classifying, describing, packaging, marking, labeling, and transporting hazardous materials, hazardous substances, and hazardous wastes are enforced by the DOT as described in Title 49 Code of Federal Regulations (CFR) Parts 171 through 178. Carrier specific requirements defined in the IATA Dangerous Goods Regulations should also be considered when preparing sample shipments conveyed by air freight providers.

4.4.7.1 Hazardous Samples

Samples containing hazardous constituents shall be considered hazardous material in transportation and transported according to DOT 49 CFR requirements. If the sample material is known or can be identified, then it shall be packaged, marked, labeled, and shipped according to the specific instructions for that material.

4.4.7.2 Radioactive Samples

Materials are classified by DOT as radioactive when the isotope specific Activity Concentration and the Exempt Consignment limits described in 49 CFR 173 are exceeded. Samples shall be screened, or relevant historical data shall be used to determine if these values are exceeded. When screening or historical data indicates that samples are radioactive, they shall be properly classified, described, packaged, marked, labeled, and transported according to DOT requirements.

Prior to shipping radioactive samples to the laboratory, the organization responsible for managing the samples shall notify the laboratory of the number and radiological level of the samples and request acceptance. This notification is conducted through a sample management organization or similar laboratory coordination group. However, the laboratory is responsible for ensuring that the applicable radioactive material license limits are not exceeded.

4.4.7.3 Holding Times and Turn-Around Times

Sample holding time is the maximum duration of time that can elapse between collection to extraction and analysis of the bulk sample. Holding times are method and matrix specific, and are identified in the project-specific SOPs, SAP, or other work control documents for each analyte or group of analytes to be determined. Exceedance of holding times may result in degradation of sample quality to the point where analytical data usability is affected.

- Sample shipment and delivery shall be coordinated with the laboratory to meet sample holding times, where applicable.
- Sample holding time begins at the time and date of collection recorded on the chain-of-custody. The sample holding times ends either when the sample is analyzed or extracted.
- The use of preservatives may extend the acceptable sample holding times. This approach can be negotiated with the regulators to support the collection of cost-effective data of known and controlled variability.
- Recommended sample holding times guidelines are provided in Appendix A. These represent the maximum generally accepted lengths of time where, under the specified preservation conditions, significant loss of analytes, or degradation of analytes is not expected to occur. Unless there is reason to believe that unique circumstances could accelerate or delay the loss or degradation of analytes in a given set of samples, the holding times in Appendix A should not be deviated from. A project team may determine the applicability of holding times based on sampling and analysis constraints, data use, or other technical criteria. These determinations shall be documented in the applicable DQO document, SAP, or in project records.

The consequences and impacts of missed holding times on data quality shall be taken into account in the sample collection planning process (Section 2.0), and actual missed holding times should be discussed or otherwise noted in the analytical data package or analytical report from the laboratory.

The turn-around time is a contractual specification, and is the amount of time from sample receipt at the laboratory to when data are reported to the client.

4.4.8 Holding Times for Highly-Radioactive Samples

Short holding times (i.e., on the order of 24 hours to one to two weeks) required for some analyses may not be achievable for highly-radioactive samples because of the increased time required to handle high radiation samples with remote handlers in hot cells. The increased logistics required to survey, transport, and screen highly-radioactive samples before analysis may also contribute to missed holding times.

4.5 DECONTAMINATION AND CLEANING OF EQUIPMENT

Clean sampling equipment shall be used during sample collection to avoid contamination of samples. Sampling equipment shall be decontaminated after use to avoid spreading contamination to non-contaminated locations and to avoid contaminating sampling personnel. SOPs shall describe how sampling equipment is to be cleaned and decontaminated. Single use sampling equipment is allowed.

4.5.1 Decontamination and Cleaning of Equipment Used to Collect Highly Radioactive Samples

Sampling equipment used for highly radioactive samples may be a one-time use process. Therefore, equipment decontamination during the collection of highly radioactive samples may not be possible. The consequences of not being able to decontaminate equipment during the collection of highly radioactive samples should be considered in the sample collection planning Process (Section 2.0).

4.6 FIELD CHANGES

Unexpected field conditions encountered during sampling may require that changes be made to the sampling requirements found in project-specific SOPs, SAP, or other work control documents. Three levels of change control are recognized that affect the level of agreement and documentation necessary to implement a field change:

- A **minor field change** is a change that has no adverse effect on the technical adequacy of the sampling activity or the work schedule associated with the approved sampling document. Minor field changes shall be documented in the field logbook. The logbook entry shall include the field change, the reason for the field change, and the names and titles of those approving the field change
- A **minor change** generally involves changes to an approved sampling document but do not affect the overall intent of the document or associated schedule. The Operable Unit Project Manager will inform the DOE Richland Operations Office Project Manager or DOE Office of River Protection Project Manager and the Regulatory Lead of the change and seek concurrence at a Unit Manager's Meeting or comparable forum. The lead regulatory agency determines if there is no need to revise the document.
- A **revision necessary** change occurs when the lead regulatory agency determines that the change requires the approved sampling document should be updated to reflect the change.

4.7 WASTE DISPOSITION

Waste materials are generated during sample collection, processing, and sub-sampling activities. The method of identification, storage, and disposition of these waste materials and unused samples shall be specified by a waste management plan when hazardous, radioactive, or mixed waste is generated.

Program and project managers shall ensure their waste management plan for the sampling and field analysis event has also addressed the return of unused sample material and/or the wastes generated from analysis inside the laboratory upon completion of the work. Information provided to the laboratory pertaining to listed waste may impact the management of the waste being returned to the program or project manager. These policies and guidelines apply to personnel who generate, handle, manage, and/or disposition waste in the field activities.

Consultation with a waste management specialist shall be considered when polices and guidelines for waste management are being developed. The waste management plan shall detail the responsibilities for waste management and handling, along with the approved disposition methods for derived wastes.

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5.0 QUALITY ASSURANCE AND QUALITY CONTROL FOR THE SAMPLING PROCESS

5.1 QUALITY ASSURANCE

Project-specific QA requirements shall be established or identified as part of the sample collection planning process. Routine QA activities in the sampling process include, but are not limited to, frequent (e.g., daily) review of site and field logs, field notebooks, and data forms; and comparison of results with the data quality requirements of the project-specific SOPs, SAP, or other work control documents. If the selection of sampling points and/or samples for more detailed examination is based on field analytical data (qualitative and/or semi-quantitative), it is necessary to review the field analytical results as well. Field documentation shall be reviewed to ensure that the proper number of field QC samples have been submitted to the laboratories.

Typical elements of these sampling QA review activities include review of:

- Documentation of the correct number and locations of the sampling points
- Proper collection, handling, and shipping of samples
- Correct completion of field records and documents
- Meeting correct data reporting requirements for the day's activities
- Ensuring sample custody requirements are met.

5.2 QUALITY CONTROL

Various types of QC samples are taken during the sample collection process to monitor the adequacy of the sampling system and the integrity of samples during their transport from the field collection point through laboratory analysis. Field-generated QC samples include blanks, duplicates, and split samples. Additionally, samples of decontamination water may be taken to evaluate the potential for cross-contamination in a decontamination or steam cleaning process. Although not directly connected to monitoring of sampling systems and sample integrity, field activities may also include the generation of field matrix spiked samples. These various types of field QC samples are discussed in more detail below.

5.2.1 Blanks

Blanks are used to detect contamination during sample handling, storage, and transportation. For liquid, sampling blanks consist of an analyte sample container filled with reagent water. For solid samples, the ideal material would be an analyte-free solid matrix, but this is not practical for most activities. Silica sand having low metallic contamination is most frequently chosen, but the source of solid blank matrix material should be addressed within the project-specific SOPs, SAP, or other work control documents. If a solid matrix is not available for a blank, reagent water may be used. Blanks shall be placed in the same storage containers and transported with the sample bottles from the associated sampling event. Blanks shall be stored at the laboratory with associated samples and processed in the same analytical batches as the associated samples.

Blank results shall be reviewed to determine if any cross-contamination occurred that could affect sample results. Sample results shall be reviewed throughout the course of the project to monitor the possible effects of any contamination detected in the blank. If contamination is detected and it is determined that ancillary sources (e.g., the field source water used for decontamination) are free of the analytes of interest, it may be necessary to monitor the field crew to ensure adherence to the procedures. If it is determined that the crews are properly following procedures, and no laboratory contamination source is determined through the result of analysis, it may be necessary to change the field procedures.

Preparation of appropriate blank samples can be difficult when remote manipulation of sampling equipment (i.e., highly radioactive contaminated) is required. Accordingly, consideration of the radioactive nature of samples must be taken in the sample collection planning process relative to field QC applicability.

5.2.1.1 Trip Blanks

Trip blanks are used to document potential contamination caused by conditions in the field during sample transport, storage, and handling. Trip blanks are prepared prior to traveling to the sampling site. Trip blanks are transported to the sampling site, not opened in the field, and shipped to the laboratory with the samples as part of the sample set. Trip blanks are primarily used for volatile organic compounds (VOCs). VOC analysis-only trip blanks are frequently identified as daily trip blanks. Trip blanks, however, may be used whenever there is concern that concentration of an analyte may be biased by contamination. Trip blanks for which more than VOC analysis is requested are referred to as full trip blanks. Full trip blanks may identify a subset of the analyses suite as defined by project-specific SOPs, SAP, or other work control documents.

One VOC trip blank shall either accompany each cooler that contains site samples for VOC analysis or shall accompany samples as specified in the project-specific SOPs, SAP, or other work control documents. Full trip blanks typically are collected at a frequency of 1 per 20 samples or as specified in the project-specific SOPs, SAP, or other work control documents.

5.2.1.2 Transfer Blanks

Transfer blanks are used to document contamination caused by conditions in the field sampling. Transfer blanks are preserved sample bottles filled at the sample collection site with high purity reagent water or silica sand that has been transported to the sampling site. This blank is sealed at the site and becomes part of the sample set sent to the laboratory.

Transfer blanks are primarily used for VOCs. VOC analysis-only transfer blanks are frequently identified as field transfer blanks. However, they may be used whenever there is concern that concentration of an analyte may be biased by contamination. Transfer blanks for which more than VOC analysis is requested are referred to as field blanks. Field blanks may identify a subset of the analyses suite as defined by project-specific SOPs, SAP, or other work control documents.

Field transfer blanks shall be prepared daily for sites sampling for VOC analysis or shall accompany samples as specified in the project-specific SOPs, SAP, or other work control documents. Field blanks typically are collected at a frequency of 1 per 20 samples or as specified in the project-specific SOPs, SAP, or other work control documents.

5.2.1.3 Equipment Blanks

Equipment blanks, also known as equipment rinsate blanks, are used as a measure of decontamination process effectiveness. They are samples of high purity reagent water or silica sand put in contact with the sampling surfaces of equipment used to collect samples prior to use of that equipment in the same environment. Equipment blanks are typically collected at a frequency of 1 in 20 samples or at the frequency as specified in the project-specific SOPs, SAP, or other work control documents. An equipment blank shall be collected from each type of sampling equipment used to ensure that the decontamination procedures are applicable to the specific equipment types. Equipment blanks shall be analyzed for the same analytes as samples collected using that equipment or as specified in the project-specific SOPs, SAP, or other work control documents.

5.2.1.4 Bottle Blanks

A trip blank not only will detect contamination during the shipping and handling of the containers, but could also serve to detect contamination from containers (i.e., function as a bottle blank), which is important if non-certified sample containers are being used. If contamination from the container is suspected, specific bottle blanks should be generated where other potential impacts (e.g., transportation and storage in the field) are minimized. Bottle blanks shall be collected at a frequency as specified in the project-specific SOPs, SAP, or other work control documents. Bottle blanks shall be analyzed for the same analytes as samples collected or as specified in the project-specific SOPs, SAP, or other work control documents.

5.2.1.5 Field Source Water Blanks

Field source water blanks are samples of source water used for decontamination and steam cleaning. This may prevent the introduction of contaminants to the site samples. Normally, there will be two types of field source water blanks: (1) a sample of the potable water used for steam cleaning, and (2) a sample of the reagent water used for decontamination. If more than one batch or lot number of reagent water is used, or if potable water is taken from more than one location, then additional field source water blanks shall be taken since these are different sources. Field source water blanks shall be collected at a frequency as specified in the project-specific SOPs, SAP, or other work control documents. Field source water blanks shall be analyzed for the same analytes as samples collected or as specified in the project-specific SOPs, SAP, or other work control documents.

If contamination is detected, a different source of water should be used.

5.2.2 Field Duplicates (Replicates)

Field duplicates provide information regarding the homogeneity of the sample matrix. Field duplicates may also provide an evaluation of the precision of the sampling and analysis process. Field duplicates are two samples that are intended to be identical and shall be collected as close as possible to the same time and same location. Each will be numbered uniquely. Unless specified differently in the project-specific SOPs, SAP, or other work control documents, for field duplicates (except for VOC analysis) the volume needed is collected and homogenized before being divided into two samples in the field. Field duplicates normally will be collected at a frequency of 5 percent to 10 percent of the samples collected per matrix or as specified in the project-specific SOPs, SAP, or other work control documents. Samples submitted for VOC analyses are not to be homogenized or split; instead, it is necessary to collect collocated samples as defined in Section 5.2.2.2.

Field duplicates shall be sent to the laboratory in the same manner as the routine site samples. They may or may not be identified to the laboratory as field duplicates. It may maximize the utility of information to submit extra samples from the field duplicates for the laboratory to use as duplicates. This will help distinguish between variability resulting from sample heterogeneity and from laboratory manipulation.

5.2.2.1 Field Split Samples

Split samples serve as inter-laboratory comparisons samples. Split samples are a variation of field duplicate samples. Split samples are intended to be identical and shall be collected as close as possible to the same time and same location. The frequency and method for collection of field split samples is directed by the project-specific SOPs, SAP, or other work control documents. Usually, twice the routine volume needed is collected, homogenized, and subsequently placed in separate, identically prepared containers, numbered uniquely, and forwarded to separate laboratories for analysis using the same method/protocol. VOC analysis splits are samples collected as collocated samples as described in Section 5.2.2.2.

5.2.2.2 Collocated Samples

This sampling protocol is used where homogenizing samples for duplicate or split samples would impact the quality of the resulting data or as specified in the project-specific SOPs, SAP, or other work control documents. Collocated samples are not homogenized. Because of the possible loss of volatile analytes when generating field duplicates, it is necessary to collect samples for VOC analysis as collocated samples. Collocated samples are independent samples collected as close as possible to the same point in space and time and are intended to be identical as practical. For liquid samples for VOC analysis, duplicate or split samples are typically collected sequentially during the sampling event. Collocated soil cores collected for VOC analyses shall be sealed immediately and shipped to the laboratory.

5.2.3 FIELD MATRIX SPIKES

Field matrix spikes provide information about potential analytical bias from degradation during shipping and storage, in addition to bias from the sample matrices. Field matrix spikes can be used to verify that the sample holding times were sufficient for the sites sampled during the course of a project. An aliquot of the sample matrix is spiked with a known concentration of target analyte(s). The spiking usually occurs during or immediately after sampling before the sample is sent to the laboratory. Establishing spiking levels may be complex but should take into account expected analyte concentrations levels in the sample matrix, expected analytical detection limits, and desired analytical decision levels. Matrix composition, spiking levels, and frequency will be as specified in the project-specific SOPs, SAP, or other work control documents.

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6.0 REFERENCES

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APPENDIX A
RECOMMENDED PRESERVATION, CONTAINER, AND
HOLDING TIME GUIDELINES

PRESERVATION, CONTAINER, AND HOLDING TIME

Parameter	Matrix	Container	Preservation ^a	Holding Time
Inorganic Analyses				
<i>Gravimetric Determinations</i>				
Residue, Filterable Total Dissolved Solids	Water	Narrow mouth Poly or Glass	Store ≤ 6°C	7 Days
Residue, Non- filterable Total Suspended Solids	Water	Narrow mouth Poly or Glass	Store ≤ 6°C	7 Days
Total Solids	Water	Narrow mouth Poly or Glass	Store ≤ 6°C	7 Days
<i>Inorganic Ions</i>				
Ammonia	Water	Narrow Mouth Poly or Glass	Store ≤ 6°C, Adjust pH to <2 with H ₂ SO ₄	28 Days
	Soil	Wide Mouth Poly or Glass	None Required	28 Days after Extraction
Bromide	Water	Narrow Mouth Poly or Glass	Store ≤ 6°C	28 Days
Chloride	Water	Same as above	Store ≤ 6°C	28 Days
Fluoride	Water	Same as above	Store ≤ 6°C	28 Days
Sulfate	Water	Same as above	Store ≤ 6°C	28 Days
Phosphate (Total)	Water	Same as above	Store ≤ 6°C	28 Days
Nitrate + Nitrite	Water	Same as above	Store ≤ 6°C	28 Days
Phosphate (Ortho)	Water	Same as above	Store ≤ 6°C	48 Hours
Nitrate	Water	Same as above	Store ≤ 6°C	48 Hours
Nitrite	Water	Same as above	Store ≤ 6°C	48 Hours
Anions (per above)	Soil	Wide mouth Poly or Glass	None	Sampling to extraction: 28 days. After Extraction Refer to Water Holding Times for Specific Anions

Parameter	Matrix	Container	Preservation ^a	Holding Time
Sulfide	Water	Wide mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$, ZnAc+NaOH to pH > 9	7 Days
	Soil	Glass	Store $\leq 6^{\circ}\text{C}$	Sampling to extraction: 28 Days; After extraction: 7 ^o Days
Metals				
Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with/without Mercury	Water	Narrow Mouth Poly or Glass	Adjust pH to < 2 with Nitric Acid	28 days/6 Months ^b
	Soil	Wide Mouth Poly or Glass	None Required	28 days/6 Months
	Other	Poly or Glass	Store $\leq 6^{\circ}\text{C}$	28 days/6 Months
Inductively Coupled Plasma Atomic Emission Spectroscopy with/without Mercury	Water	Narrow Mouth Poly or Glass	Adjust pH to < 2 with Nitric Acid	28 days/6 Months
	Soil	Wide Mouth Poly or Glass	None Required	28 days/6 Months
	Other	Poly or Glass	None Required	28 days/6 Months
Mercury Only	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with Nitric Acid	28 days
	Soil	Wide Mouth Poly or Glass	None Required	28 days
	Other	Poly or Glass	None Required	28 days
Toxicity Characteristic Leaching Procedure (TCLP) Extraction: Metals	Soil	Wide Mouth Amber Glass	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 28 days/6 Months Extraction to Analysis: 28 days/6 Months
	Other	Amber Glass	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 28 days/ 6 Months Extraction to Analysis: 28 days/ 6 Months

Parameter	Matrix	Container	Preservation ^a	Holding Time
Dissolved Metals (With or Without Mercury)	Water	Narrow Mouth Poly or Glass	Filter prior to pH adjustment to < 2 with Nitric Acid	28 days/6 Months
Misc. Inorganic				
Alkalinity	Water	Narrow mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$	14 Days
	Soil	Wide Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$	14 days
Chemical Oxygen Demand	Water	Narrow Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$, Adjust pH to <2 with H_2SO_4	28 Days
	Soil	Wide Mouth Poly or Glass	Store @ $\leq 6^{\circ}\text{C}$	28 days
Cyanide (Total)	Water	Narrow Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$, Adjust pH to >12 with 50% NaOH. If oxidizing agents present, add 5 ml 0.1 N NaAsO_2/L or 0.06 g ascorbic acid/L.	14 Days
	Soil	Wide Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$	14 Days
pH	Water	Narrow Mouth Poly or Glass	None Required	Analyze Immediately
	Soil	Wide Mouth Poly or Glass	None Required	Analyze Immediately
Hexavalent Chromium	Water	Narrow Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$	24 Hours
	Soil	Wide Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 30 Days Extraction to Analysis: 7 Days

Parameter	Matrix	Container	Preservation ^a	Holding Time
Specific Conductivity	Water	Narrow Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$	28 Days
Flash Point	Water	Narrow Mouth Poly or Glass	Store $\leq 6^{\circ}\text{C}$	None
Total Halides	Other	Poly or Glass	Store $\leq 6^{\circ}\text{C}$	28 Days
Organic Analyses				
<i>PCBs</i>				
PCBs	Water	Narrow Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	1 Year
	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	NA
	Other	Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	NA
<i>Semivolatile Organics Analysis (SVOAs)</i>				
SVOAs	Water	Narrow Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$ (if residual Cl_2 , add 3 ml 10% sodium thiosulfate/gal of sample)	Sampling to Extraction: 7 Days Extraction to Analysis: 40 Days
	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 14 Days Extraction to Analysis: 40 Days

Parameter	Matrix	Container	Preservation ^a	Holding Time
TCLP Extraction: SVOAs	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to TCLP Extraction: 14 Days TCLP Extract to Liquid Extract: 7 Days Liquid Extract to Analysis: 40 Days
	Other	Amber Glass with Teflon lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to TCLP Extraction: 14 Days TCLP Extract to Liquid Extract: 7 Days Liquid Extract to Analysis: 40 Days
Washington Department of Ecology Total Petroleum Hydrocarbons- Diesel	Water	Narrow Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$ pH to < 2 with HCl	Sampling to Extraction: 7 Days Extraction to Analysis: 40 Days
	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 14 Days Extraction to Analysis: 40 Days
Polynuclear Aromatic Hydrocarbons by 8310	Water	Narrow Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	7 days
	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	14 days
<i>Pesticides and Herbicides</i>				
Pesticides	Water	Narrow Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 7 Days Extraction to Analysis: 40 Days
	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 14 Days Extraction to Analysis: 40 Days

Parameter	Matrix	Container	Preservation ^a	Holding Time
TCLP Extraction: Pesticides	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to TCLP Extraction: 14 Days TCLP Extract to Liquid Extract: 7 Days Liquid Extract to Analysis: 40 Days
Herbicides	Water	Narrow Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 7 Days Extraction to Analysis: 40 Days
	Soil	Wide mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 14 Days Extraction to Analysis: 40 Days
TCLP Extraction: Herbicides	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to TCLP Extraction: 14 Days TCLP Extract to Liquid Extract: 7 Days Liquid Extract to Analysis: 40 Days
<i>Phenols</i>				
Phenols	Water	Narrow Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 7 Days Extraction to Analysis: 40 Days
	Soil	Wide mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	Sampling to Extraction: 14 Days Extraction to Analysis: 40 Days

Parameter	Matrix	Container	Preservation ^a	Holding Time
VOAs				
VOAs	Water	40 ml Amber Glass VOA Vial with TEFLON Lined Septum Lid	Store $\leq 6^{\circ}\text{C}$ (if free Cl_2 add 4 drops of 10% sodium thiosulfate), adjust pH to < 2 with HCl	14 Days
	Soil –Low Level	See Above	Store frozen	14 Days
	Soil – High Level	See Above	Methanol, Store $\leq 6^{\circ}\text{C}$	14 days
	Other	See Above	Store $\leq 6^{\circ}\text{C}$	14 Days
TCLP Extraction: VOAs	Soil	Wide Mouth Amber Glass with TEFLON Lined Lid	Store $\leq 6^{\circ}\text{C}$	Sampling to TCLP Extraction: 14 days TCLP Extraction to Analysis: 14 days
WTPH-G	Water	40 ml Amber Glass VOA Vial with TEFLON Lined Septum Lid	Store $\leq 6^{\circ}\text{C}$, adjust pH to < 2 with HCl	14 Days
	Soil	Amber Glass with TEFLON Lined Septum Lid	Store $\leq 6^{\circ}\text{C}$	14 Days
Misc. Organic				
Polar Organic Molecules by Method EPA 8015	Water	40 ml Amber Glass VOA Vial with TEFLON Lined Septum Lid	Store $\leq 6^{\circ}\text{C}$, adjust pH to < 2 with HCl	14 Days
	Soil	See Above	Store @ $\leq 6^{\circ}\text{C}$	14 Days

Parameter	Matrix	Container	Preservation ^a	Holding Time
Oil And Grease	Water	Narrow Mouth Glass	Store $\leq 6^{\circ}\text{C}$, adjust pH to < 2 with HCl	28 Days
	Soil	Wide Mouth glass	Store @ $\leq 6^{\circ}\text{C}$	28 Days
Total Organic Halides	Water	Narrow Mouth Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$, Adjust pH to < 2 with H_2SO_4	28 Days
	Soil	Wide Mouth Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	28 Days
Extractable Organic Halides	Soil	40 ml Glass VOA Vial with TEFLON Lined Septum Lid	Store $\leq 6^{\circ}\text{C}$	28 days
Total Organic Carbon	Water	Narrow Mouth Amber Glass with TEFLON Lined Lid	Store $\leq 6^{\circ}\text{C}$, Adjust pH to < 2 with H_2SO_4 or HCl	28 Days
	Soil	Wide Mouth Amber Glass with TEFLON lined lid	Store $\leq 6^{\circ}\text{C}$	28 Days
Radiochemical Analyses				
Total Alpha/Beta by Liquid Scintillation	Water	Narrow Mouth Poly or Glass	Adjust pH to < 2 with HNO_3	NA ^c
	Soil	Wide Mouth Poly or Glass	None	NA
	Other	Poly or Glass	None	NA
Gross Alpha/Beta (Plate Count)	Water	Narrow Mouth Poly or Glass	Adjust pH to < 2 with HNO_3	NA
	Soil	Wide Mouth Poly or Glass	None	NA
	Other	Poly or Glass	None	NA

Parameter	Matrix	Container	Preservation ^a	Holding Time
Americium/ Curium by Alpha Energy Analysis (AEA)	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
	Soil	Wide mouth Poly or Glass	None	NA
	Other	Poly or Glass	None	NA
Carbon-14	Water	Narrow mouth Poly or Glass	None	NA
	Soil	Wide Mouth Poly or Glass	None	NA
Plutonium Isotopic by AEA	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
	Soil	Wide Mouth Poly or Glass	None	NA
	Other	Poly or Glass	None	NA
Uranium Isotopic by AEA	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
	Soil	Wide mouth Poly or Glass	None	NA
	Other	Poly or Glass	None	NA
Gamma Energy Analysis (GEA)	Water	Square Poly	Adjust pH to <2 with HNO ₃	NA
	Soil	Square Poly	None	NA
	Other	Square Poly	None	NA
Iodine - 129	Water	Narrow Mouth Glass	None	NA
	Soil	Wide mouth Poly or glass	None	NA
Neptunium - 237	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
	Soil	Wide Mouth Poly or Glass	None	NA
Nickel-63	Water	Narrow Mouth Poly or Glass	None	NA
	Soil	Wide Mouth Poly or glass	None	NA

Parameter	Matrix	Container	Preservation ^a	Holding Time
Radium -228	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
Radium-226	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
	Soil by AEA	Wide Mouth Poly or Glass	none	NA
	Soil by GEA	Wide Mouth Poly or Glass	None	NA
Strontium - 90	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
	Soil	Wide Mouth Poly or Glass	None	NA
	Other	Wide Mouth Poly or Glass	None	NA
Technetium - 99 by Liquid Scintillation	Water	Narrow Mouth Glass	Adjust pH to <2 with HCl	NA
	Soil	Wide Mouth Poly or Glass	None	NA
Technetium - 99 by ICP-MS	Water	Narrow Mouth Poly or Glass	None Required	NA
Tritium	Water	Narrow Mouth Glass	None	NA
	Soil	Wide Mouth Glass	None	NA
Total Uranium by Kinetic Phosphorescence Analysis	Water	Narrow Mouth Poly or Glass	Adjust pH to <2 with HNO ₃	NA
	Soil	Wide Mouth Poly or Glass	None	NA

^a For preservation identified as Store @ $\leq 6^{\circ}\text{C}$, the sample should be protected against freezing unless it is known that freezing will not impact the sample integrity.

^b For metals analysis, "28 days/6 Months" holding time defines 28 days for mercury, 6 months for all other metals.

^c Holding times for short half-life radionuclides shall be < 6 half-lives. Longer lived radionuclides should be analyzed within 6 months.

NOTES:

- Under the heading "Container" the term Poly stands for EPA Clean Polyethylene Bottles.
- The matrix "Other" includes, but is not limited to:
 - Air/gases
 - Asbestos or suspect asbestos containing solids

- Ash
 - Biota
 - Concrete
 - Fuel
 - Oil
 - Sludge
 - Slurries
 - Wipes
 - Other solids and liquids.
3. The information in this table does not represent EPA requirements, but is intended solely as guidance. Selection of container, preservation techniques, and applicable holding times should be based on the stated project-specific DQOs.