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## **1.0 INTRODUCTION**

This volume, used with Volume 1, “Administrative Requirements,” sets forth the quality assurance (QA) and quality control (QC) requirements that govern analytical work performed in the field. Field analytical techniques are often qualitative or semi-quantitative analytical methods that can be used to provide a quick determination of the presence, magnitude, or absence of a contaminant. This minimizes the expense and extended turnaround time required to analyze samples in a laboratory. Because the QC and analytical sophistication are not controlled to the same extent as laboratory analysis, a cross-section of additional samples may need to be submitted to a fixed laboratory for confirmatory quantification. Fixed laboratory methods are further described in Volume 4.

As a matter of convention in this document, the measurement of samples in the field may be performed by field screening techniques or field-testing techniques. The data quality objective (DQO) process should dictate the type of measurement system that will be used. A graded approach is recommended to determine the specific field analytical method and applicable QC based upon cost considerations and the decision risk associated with use of the data, and is further discussed in Section 5.1.1.

### **1.1 FIELD SCREENING**

Field screening procedures are performed directly on the sample or media in the field. A variety of field screening methods are available and the project must be cautious and clearly define and control the use of any data generated. The chance of false positives and false negatives must be known and adequate QC must be demonstrated to ensure acceptance of the procedures for use by the decision maker or regulatory authority. Measurements are usually acceptable, as long as the method detection limit is below the action levels or regulatory threshold limits. The best results for field screening methods are obtained when the contaminant is known and the technique is acceptable for use under the environmental conditions that exist at the site.

### **1.2 FIELD TESTING**

Field testing procedures are performed on samples collected in the field and generally analyzed in a field laboratory located at or near the sample collection point. Field analytical techniques many times use the same analytical methods as a fixed laboratory. Data generated are many times used for compliance and must meet similar QC standards as a fixed laboratory and, therefore, require environmental and site conditions to be recorded to ensure that data comparability is based on test conditions similar to those for the fixed laboratory. The level of quality required for the data is determined by the project using the DQO process.

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## **2.0 QUALITY ASSURANCE REQUIREMENTS**

The QA objectives and requirements provide a set of recognized parameters to monitor and quantify performance of an analytical measurement. At Hanford, QA requirements to be applied are considered in the context of the overall objectives of the project that will use the data. The project and associated DQOs/requirements are derived through using the DQO process. The DQOs that result from the process are then translated into a sampling and analysis plan (SAP) for the project.

This section provides a brief overview of field quality requirements for the field analytical organization. For this document, the field analytical organization is defined as any company or group providing radiological, organic, and/or inorganic field screening or field-testing services in support of environmental decision-making.

### **2.1 DATA QUALITY OBJECTIVES**

After developing project DQOs/plans, the client or data user discusses basic information about the nature of the sample(s) and the intended use of the data with the field analytical organization prior to sample collection. As a minimum, the field analytical organization and the client shall agree upon the method performance criteria such as precision, accuracy, and sensitivity for all analyses (e.g., instrument detection limit [IDL], minimum detection level [MDL], and practical quantitation limit [PQL]). For field organizations performing field screening (i.e., analysis of samples in place, such as real-time air monitoring at set locations, radiological surveys of structures, equipment, or the environment), the client will identify critical locations where samples or measurements must be obtained.

The field analytical organization shall have a system to notify and explain any unique project requirements to all staff performing work for the client. Unique requirements are those that differ from the procedures described in this document and in the field analytical organization's standard operating procedures.

The field analytical organization shall notify the client when situations occur that were not anticipated in the SAP. These anomalies or nonconformances may result in plan changes by the project team. To the extent necessary, the field organization should make staff and information available to support the team to resolve technical issues in a manner that is acceptable to the client or data user.

## **2.2 CLIENT DATA QUALITY REQUIREMENTS**

Data quality requirements are most frequently listed in the SAPs, which include a field sampling plan (FSP), quality assurance project plan (QAPjP), and other planning documents. The quality requirements shall be mutually understood and agreed upon by the field analytical organization and the client prior to start of the field activity.

The client is responsible for ensuring that access is available to the sample locations and, to the extent possible, that adequate sample material is available to meet project requirements. This can be best achieved if the client includes appropriate field screening and field-testing professionals on the planning team. The field analytical organization(s) providing field screening and/or field testing is/are responsible for using proper field analytical procedures and protective sample handling protocols. The field analysis organization and the client share responsibility for selecting appropriate field analytical techniques.

Five parameters (i.e., precision, accuracy, completeness, comparability, and representativeness) are most often used to determine field screening and field-testing data quality requirements. These parameters are defined in Volume 1, Appendix A, "Glossary." Typically these five parameters (or a subset of these) determine data quality and usability and should be considered when selecting a field screening or field-testing technique.

### **3.0 SYSTEMS QUALITY ASSURANCE**

A number of systems must be in place and operational when field screening and/or field testing are conducted. These systems must function properly to produce and document the level of quality in the final product: administrative systems, software systems, technical systems, and physical facility systems. The systems shall be described in the field analytical organization's quality management plan or project QAPjP.

#### **3.1 ADMINISTRATIVE SYSTEMS**

Volume 1 provides the requirements for administrative systems that support field analytical activities. The field analytical organization should refer to the following sections in Volume 1 for implementation of a QA requirements program:

- Organizational structure and responsibility shall be defined (Section 2.0).
- Personnel qualification and training requirements shall be described (Section 3.0).
- Processes for procedure development, revision, and control shall be provided (Section 4.0).
- Corrective actions and quality improvement program shall be in place (Section 5.0).
- Documents and quality records shall be controlled and managed (Section 6.0).

Additional administrative requirements specific to field screening and field testing are further described in the following subsections.

##### **3.1.1 Documented Quality System**

The field analytical team shall establish and maintain a quality system appropriate to the type, range, and volume of sampling, calibration, and/or testing activities. The elements of this system shall be documented in a QA plan. The QA document shall be available, as necessary, for use by the field sampling personnel, analytical personnel, data validators, and the client.

Documentation shall include and/or refer to the following:

- Procedures or instructions for measuring data precision, accuracy, representativeness, comparability, and completeness
- Procedures or instructions for field operations such as sampling, special processes, purchased items and services, equipment maintenance, instrument calibration and use, field analyses, waste verification, geophysical surveys, and other site characterization activities
- Methods to be used in data reduction, validation, and reporting
- Procedures for corrective action, nonconformance control, identification and control of items, and QC reports to management

- A training and qualifications program to ensure that field sampling/analytical personnel in all positions have been trained and qualified in their specific area of work and in the QA procedures associated with their work
- Document control procedures to ensure that appropriate versions of documentation are being used
- Project interfaces and organizational structure

### **3.2 SOFTWARE SYSTEMS**

General requirements of software systems are discussed in Volume 1, Section 7.0. Due to variances in field portable equipment, the field analytical team shall ensure that data generated from instrumentation using a software system are backed up and/or downloaded on a regular basis. Any software configuration will require acceptance testing prior to use in the field.

### **3.3 TECHNICAL SYSTEMS**

Technical systems ensure that the techniques are applicable and properly applied. These systems may include sample exchanges, standards programs, control of standards and reagents, data reduction and reporting, data verification and validation, and technical audits/assessments. Procedures for documenting these applicable systems shall be established. Volume 1, Section 8.0, "Procurement Controls," describes the requirements for procured items and services. Volume 1, Section 9.0, "Equipment/Preventative Maintenance," describes the requirements for preventative maintenance of field screening and field-testing equipment.

#### **3.3.1 Field Assessments**

During the actual performance of field activities, in-process self-assessments or surveillances should be performed according to the requirements established in Volume 1, Section 10.2. At a minimum, one field assessment should be performed in each area of field activity within a defined period-of-time (e.g., annually) or within the lifetime of the project (i.e., if the project duration is less than one year).

The audit/self-assessment/surveillance of field analytical activities should be selected from the following subjects:

- Equipment – Equipment, used for collection, measurement, and testing, should meet the applicable standards (e.g., American Society for Testing and Materials [ASTM]) or have been evaluated as acceptable and valid in accordance with the procedures, requirements, and specifications



- Verification of analysis activities – Analysis activities that are the elements of the analysis program will be verified for compliance with the applicable technical and quality standards, specifications, and SAP requirements. The elements to be verified include, but are not limited to, the following:
  - Implementation of QA requirements
  - Qualification of personnel
  - Identification, control, and storage of samples, standards, project documents, and generated data
  - Implementation of methods or procedures conforming to applicable specifications and SAP/work plan requirements
  - Documentation and verification of conditions, observations, and corrective actions taken
- Completeness of records – To determine if records are complete, the following will be examined:
  - Measurements required by the project have been collected
  - Adequate records exist for each sample (or measurement) set and the associated QC samples
  - Specified procedures have been used and changes have been noted according to the established procedures
  - Anomalous field data are identified and appropriately qualified.
- Evaluation of data with respect to control limits – Corrective action reports will be examined to determine whether samples associated with out-of-control events are identified in a written report of the sampling activities.
- Review of holding time data – Sample holding times will be examined in comparison to the holding times required by the project. Any samples associated with deviations from holding time requirements are identified in a written record of the activities.
- Implementation and effectiveness of corrective actions – Corrective action reports will be examined to determine whether analysis activities associated with findings from previous audits/self-assessments/surveillances of similar activities were adequately addressed and corrected.
- Completeness and accuracy of reports to clients – The reports to clients will be examined to confirm that all data are accurately reported. This includes QC data reports and its potential impact on data accuracy.

### 3.3.2 Field Documentation and Record keeping

Volume 1, Section 6.0, "Documents and Quality Records," describes the general requirements for field documents and records.

Field activity record keeping and document control shall be specified, prepared, and maintained and should include the following, as applicable:

- Materials quality documentation (e.g., certifications), which concerns the quality of material used for field activities, shall be retained with project files
- Chain of custody
- Field measurement data, calibrations, and analytical results
- Logbooks, log forms, or equivalent
- Site-specific FSP, QAPjP, or work plan
- Any sampling plan modifications or changes, field analytical procedural modifications, deviations, or substitutions, as well as the reason for the change.

All documentation must be completed in indelible ink. Corrections must be marked with a single line, dated, and initialed. Handwritten documents such as logbooks and forms must be legible. All logbooks are individually identified with a control number. The field logbooks are usually considered as the master record that documents the sampling event and the operation and maintenance of all field analytical testing equipment. Requirements for records associated with electronic systems and databases used as part of a field screening or field-testing measurement system are further discussed in Section 6.0.

As a minimum, the following entries will be made in the field analytical organization's documentation for each field analytical activity:

- Names of personnel involved in the field activity
- Signature of person making an entry (the printed name of the person should be indicated at least once per logbook/electronic record by the person's signature)
- Field observations
- Instrument calibration information or reference
- Equipment identification numbers (if applicable)
- Field decontamination of equipment and personnel (if applicable)

- Field problems, solutions, and corrective actions
- Sample identification, identification numbers, date, time, preservative (if required), and analysis
- Field measurement data, types of method, types of QC samples used, and field screening data
- Field measurement location
- Pertinent weather or environmental conditions that may affect data
- Lot numbers of the sample collection containers used
- Radiological screening information
- Variances and field changes

Field logbooks and other field information forms will be reviewed by the team lead, the person in charge, or the site's appointed QA representative within a reasonable timeframe to ensure that all applicable and necessary information is present to support sampling and data quality.

### **3.4 PHYSICAL FACILITY SYSTEMS**

#### **3.4.1 Physical Layout**

The physical layout of the site must allow the deployment of a mobile laboratory, installation of monitoring equipment and appropriate power source, or access by personnel to deploy hand-held monitoring equipment. The location and installation of monitoring equipment must ensure that the sampling component of the monitor is measuring a representative sample.

#### **3.4.2 Facility Design and Maintenance**

Proper facility design and maintenance can help to alleviate problems associated with data generation. The following issues, as a minimum, shall be addressed:

- Ventilation, with air exchange rates and pressure differential between work area, suitable working environment (e.g., lighting and temperature control), stable power sources, and radio frequency shielding
- Adequate space for field analytical activities so the activities do not adversely affect analyses (including solvent, standards, reagent, and waste storage, in addition to other work areas)
- Specialized equipment, such as a laboratory hood or high-efficiency particulate air filtration system, where required

- Water purification
- Preventive maintenance schedules for equipment
- Proper maintenance to prevent contaminating vacuum systems
- Storage of gases

### **3.4.3 Field Analytical Laboratory Safety**

Each mobile/field analytical laboratory shall maintain operations in a safe manner and shall examine activities to determine potential hazards. All work shall adhere to the applicable corporate safety, industrial hygiene, and radiological control procedures. When field analysis is performed, portable eyewash stations and appropriate spill kits shall be located near the point of analyses. Entry into the mobile laboratories shall be limited to approved personnel or individuals escorted by approved personnel. Whenever a mobile laboratory is performing analysis on radiologically contaminated samples, appropriate radiological working procedures shall be in place, as well as personnel monitoring and, in some cases, air monitoring. Entrances to the mobile facility shall have the appropriate signs on the entry door regarding personnel protective clothing and radiological conditions/controls.

### **3.4.4 Waste Management**

Waste produced from field analysis may include sample media, calibration standards, syringes, empty sample containers, waste glassware, paper towels, gloves, and other expendable items. Consideration must be given to reagents used to clean syringes or glassware because used methanol, for example, is a listed hazardous waste.

The handling and disposal of radioactive waste and hazardous waste generated by field analytical methods shall comply with all Federal, state, and local requirements. Procedures shall incorporate controls that minimize the generation of waste and maximize the concentration, recovery, and recycling of waste products to the extent economically practical. Site-specific requirements and responsibilities for waste handling and disposal of field analytical waste shall be specified in the SAP or other project documentation.

Prior to selecting a field analytical method, an evaluation shall be made to determine the characteristics and volume of any waste that may be generated as a result of the analysis. The evaluation shall include a preliminary determination of whether the analysis will cause the generation of any hazardous, radioactive, and/or mixed waste. Additional consideration of potential listed waste is required, if applicable. Field screening and field testing methods shall be selected that minimize or do not create hazardous waste.

## 4.0 CALIBRATION

In general, the performance of measurement systems is controlled through calibration and monitored by continuing verification of calibration. This section describes the calibration activities associated with calibration requirements for field measurement systems and the specifications for standards that are used for calibration.

The procedures used to calibrate instruments for radiological, inorganic, and organic analyses are based on the operating characteristics of the instruments used in the measurement process. The frequency of calibration is directly related to the stability of the instruments, particularly the detector systems that are employed. Radiological detectors are typically extremely stable over time and may only require calibration annually, semi-annually, or when required. Some types of detectors used for organic analysis are much less stable and require calibration prior to each use. Manufacturer's instructions may be used to calibrate field instruments and should be incorporated into operating procedures whenever possible. Documentation of calibration procedures (or references) and calibration results should be recorded in field documentation. Volume 4, Section 4.0 may also be used in the calibration of instruments for analysis of similar types of chemical constituents (i.e., radionuclide, organic, and inorganic).

### 4.1 CALIBRATION OF FIELD SCREENING AND FIELD TESTING SYSTEMS

Calibrations must be performed as follows:

- Prior to initial use of a field analytical measurement system
- At the frequency recommended by the manufacturer or procedure, or as required by regulations
- Upon failure to meet specified QC criteria.

#### 4.1.1 Initial Calibration

The initial calibration verification for field analytical measurement systems checks the accuracy of the calibration and the standards used for that purpose. A level of independence shall exist between the materials used for calibration and for initial calibration verification when such materials are available. When an independent source is not available, the field analytical organization should attempt to purchase an alternate lot of the same material. Some field analytical measurement systems may be calibrated electronically as specified by the instrument manufacturer.

#### **4.1.2 Continuing Calibration Verification**

The continuing calibration verification (CCV) confirms that the original calibration is within acceptance criteria over time. This standard shall be from the same source as that used for either calibration or initial calibration verification.

#### **4.2 CALIBRATION RECORDS**

The field analytical organization's documentation shall include a record of calibration data for all methods and instruments. Calibration records (i.e., initial calibration and CCV) shall include the raw calibration data, identification of the standards used, associated reports, date of analysis, and analyst's name or initials, at a minimum. Calibration data shall be traceable to the standards used. All samples analyzed shall be traceable to the calibration under which the results were produced. Sample analysis can only proceed when measurement systems are accurately calibrated. All calibration records shall be maintained in accordance with Volume 1, Section 6.0.

#### **4.3 BALANCES, THERMOMETERS, AND PIPETTES**

Calibration records of measurement devices such as analytical balances and thermometers for critical mass and temperature measurements shall be maintained. All analytical balances shall be calibrated annually, at a minimum, by an approved metrology organization. An approved metrology organization is one that has been evaluated and selected on the basis of specified criteria that are consistent with industry standards for the calibration of balances. These records shall contain the date of calibration, the identity of the person performing the calibration, the identity of the device or serial number, and the date that the calibration expires. This information shall be affixed on or near the balance. The field analytical organization shall review the calibration data for compliance with the specified criteria. Acceptable balance calibration shall be verified and documented daily when the devices are in use. The accuracy of thermometers and thermocouples used for critical temperature measurements (e.g., refrigerator temperature for sample storage or total dissolved solids analysis) shall be verified annually by comparing readings of such devices with the readings of a National Institute of Standards and Technology (NIST)-traceable factory-certified thermometer. If radiological conditions limit this capability, then the thermometer should be checked at the ice point. The NIST-traceable factory-certified thermometer shall be re-verified at a specified frequency.

Pipettes used for critical measurements shall be verified to ensure acceptable performance.

#### **4.4 GENERAL REQUIREMENTS FOR CALIBRATION STANDARDS**

The following standard specifications shall be used, unless otherwise specified in Section 5.1.2 of this volume.

Standards used for calibration of measurement systems shall be traceable to a nationally or internationally recognized standard agency source or measurement system, if available.

A program for verifying and documenting the accuracy and traceability of all working standards against appropriate primary grade standards or the highest quality standards available shall be routinely followed. Complete documentation of the standards shall become part of the project's permanent record.

Standards used for calibration shall be accompanied by a certificate or record that includes the vendor, lot number, purity, date of preparation and/or expiration, and concentration or activity of the standard material. At a minimum, the following information shall be maintained for standard preparations and, if possible, placed on the label:

- Name of preparer
- Date prepared
- Standard identification number
- Parent standard identification number
- Dilution performed
- Dilution solvent
- Final concentration or activity
- Expiration date or shelf life (if applicable)

Standards used as radiation check sources shall be traceable and of known quality. When recognized standard material is unavailable or its purchase is impractical, the field analytical organization should attempt to purchase standard material from a reliable source. The field analytical organization shall have procedures in place to determine the acceptability of such materials.

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## 5.0 QUALITY CONTROL

The purpose of this section is to provide QC requirements for field screening and field-testing methods to ensure that the results are accurate and that the sources of uncertainty are identified and controlled. Both QC and QA are initiated at the start of a project and are integrated into all field analysis activities as the DQOs are developed.

Field screening and field testing shall be performed by trained individuals in accordance with approved written procedures and with properly calibrated instruments that are sensitive to the suspected contaminant. In addition, the field analytical methods must also incorporate QC samples throughout the sampling and analysis processes to provide data for evaluating the effectiveness of analytical processes. The QC samples permit assessment of the quality of field-generated environmental data. The information gained from the QC assessment can then be used, where necessary, to implement corrective actions during the field sampling and analytical process or to improve processes for future application.

The field analytical group responsible for field screening and field testing must ensure that the QC applied to a given scope of work is capable of meeting client objectives for precision and accuracy, or the field analytical group must negotiate alternative requirements.

### 5.1 OVERVIEW OF QUALITY CONTROL PROCEDURES

The HASQARD QA/QC program is based on a variety of QC samples. The results are used to measure method performance, guide real-time corrective action, and document the reliability of analytical data. These QC procedures apply to both field screening and field testing; however, the requirement for specific QC procedures may be modified by the individual project. All measurement systems must be responsive to the target analyte in known manner (frequently referred to as “calibration”) and this response must be confirmed at intervals during the analytical process (i.e., continuing calibration verification). All other QC procedures are applied as often as needed to ensure that variables known to effect method performance are in control during the analysis. The QC measurements help to assess method performance in terms of selectivity, precision, accuracy, and sensitivity.

The major variables affecting field analytical performance and subject to QC are as follows:

- Type of measurement system (instruments) used
- Analytical method used
- Sample matrix
- Target analytes or radionuclides
- Time
- Variable environmental field conditions (e.g., temperature extremes)
- Differences between operators
- Differences between field analytical organizations

The HASQARD QC requirements establish controls to ensure a documented degree of precision, accuracy, and reproducibility. Based on the field screening and field testing design, QC samples allow the data user (working in cooperation with the field analytical organization) to control and document analytical performance based on data needs, the characteristics of the site, and the analytical resources available.

### **5.1.1 Field Screening and Field Testing Requirements**

Tables 5.1 and 5.2 establish frequencies and acceptance levels for QC samples for selected field testing and field screening instrumentation. The tables also present a brief definition and description of the purpose of each sample, acceptance criteria, and frequency requirements. The tables are organized according to instrument types. The QC samples that are applicable to a specific class of analytes (i.e., radiological, organic, and inorganic) are also indicated.

The performance of the instrument (measurement system) is considered under ideal conditions and/or with ideal matrices and target analytes. The possible impacts from the method (e.g., sample preparation, reagents, temperature, and execution) and the impacts of other constituents or the sample matrix in the field sample in the measurement of the target analyte are addressed by the required/recommended QC samples. The specific QA/QC criteria that are applied to any set of analyses shall be decided by the project team during the planning process and communicated to the field analytical organization in work authorization documents.

### **5.1.2 Standards**

After the stabilization and calibration of the analytical equipment in accordance with the analytical method, the calibration shall be verified with the analysis of an independent verification standard. A qualified person other than the analyst should prepare an acceptable independent standard, or the standard can be purchased, as a certified pre-made standard from a vendor. Laboratory-prepared verification standards are preferably made with chemicals from a different source rather than the chemicals used in the calibration standards. If the independent verification fails to pass the method-specific acceptance criteria, the calibration sequence must be repeated.

The CCV demonstrates instrument performance or stability (e.g., monitors calibration drift) during the analysis. The CCV shall be performed at the beginning and at the end of the analysis. The CCV can be one of the standards from the initial calibration curve. The CCV must meet the method-specific acceptance criteria or recalibration must be performed. Any samples not bracketed by compliant verification standards shall be reanalyzed.

**Table 5.1. Field Screening Instrumentation Quality Requirements.**

<b>Instrument</b>	<b>Use</b>	<b>Detection Limits</b>	<b>Calibration</b>	<b>QC Requirements</b>	<b>QC Criteria</b>	<b>Comments</b>
Total organic vapor meters	Sample screening for volatile compounds; soil gas screening.	PID - 0.2 ppm, benzene.	Initial prior to use.	Verification check before and end of use.	±30%	DQO action levels; PID cannot detect compounds with ionization potential >11.
Indicator kits (colormetric); immunoassay	Field screening for a variety of analytes; gross quantification.	Varies by kit; follow manufacturer's instructions.	Initial by manufacturer; check color response with level of analyte.	N/A	N/A	Check expiration dates and storage requirements; interference from other compounds. DQOs may require additional QC.
Gas chromatograph	Sample analysis of known volatile organic compounds.	Highly dependent upon detector; method detection limit study required; must be defined prior to use.	Three-point initial.	Continuing checks to bracket the analytical run. Minimum 1 blank/day.	±50%	Method detection limits must be supplied and meet DQOs.
pH meter	Field screening (water and soil).	N/A	Two-point with standards within the expected range.	Verification checks before and after use.	Accuracy to ±0.5 pH units	Check pH calibration based on manufacturer's recommendations.
Conductivity meter	Determine conductivity of water.	N/A	One-point in 0.01N KCL solution.	N/A	N/A	Follow manufacturer's instructions.
Dissolved oxygen meter	Determine amount of dissolved oxygen in water.	N/A	Follow manufacturer's instructions.	N/A	N/A	Follow manufacturer's instructions.
Turbidity meter	Determine turbidity of water.	0.1-1 NTU	Follow manufacturer's instructions.	N/A	N/A	Follow manufacturer's instructions.
Temperature probe/ thermometer	Measure temperature of samples.	N/A	Calibrate against NIST thermometer, yearly or more frequently depending on stability of device.	N/A	N/A	--

**Table 5.1. Field Screening Instrumentation Quality Requirements.**

<b>Instrument</b>	<b>Use</b>	<b>Detection Limits</b>	<b>Calibration</b>	<b>QC Requirements</b>	<b>QC Criteria</b>	<b>Comments</b>
G-M tube	Surveying.	Relatively high detection limit.	Initial and after repair or major maintenance or if system control cannot be established.	Source check daily before and after use. Daily background measurement.	Within $\pm 20\%$ of source activity.	--
Phospor alpha detector	Survey for alpha contamination.	Project-specific.	Initial and after repair or major maintenance or if system control cannot be established.	Source check daily before and after use. Daily background measurement.	Within $\pm 20\%$ of source activity.	--
Plastic scintillator	Survey for beta contamination.	Project-specific.	Initial and after repair or major maintenance or if system control cannot be established.	Source check daily before and after use. Daily background measurement.	Within $\pm 20\%$ of source activity.	--
Dual phosphor scintillator	Survey for alpha and beta contamination simultaneously.	Project-specific.	Initial and after repair or major maintenance or if system control cannot be established.	Source check daily before and after use. Daily background measurement.	Within $\pm 20\%$ of source activity.	--
Sodium iodide (NaI) scintillator	Surveying for gamma-emitting radiation, some radionuclide identification.	Project-specific.	Calibration for the energy field to be measured or site-specific calibration factors should be developed.	Source check daily before and after use. Daily background measurement.	Within $\pm 20\%$ of source activity.	--
Intrinsic germanium (HPGe) solid-state detector	Radionuclide identification through gamma analysis.	Project specific.	Initial and after repair or major maintenance or if system control cannot be established.	Source check daily before and after use. Daily background measurement.	Within $\pm 20\%$ of source activity.	Can identify and quantify concentrations of gamma emitting radionuclides with proper calibration or algorithms. DQOs may require additional QC.

N/A = not applicable

**Table 5.2. Field Testing Instrumentation Quality Requirements.**

Instrument	Use	Detection Limits	Calibration	QC Requirements	QC Criteria	Comments
Gas chromatograph	Sample analysis of known volatile organic compounds.	Highly dependent upon detector; generally parts per billion; method detection limit study required; must meet DQOs.	Three-point initial, must be within two standard deviations.	Continuing checks to bracket the analytical run.	±30%	Method detection limits must be supplied and meet DQOs. Recommend duplicates run at a frequency of 1 per 20 samples. DQOs provide additional QC.
				Minimum of 1 blank/day.	<3 X MDL	
X-ray fluorescence	Sample analysis of known inorganic compounds.	Varies widely among instruments, lowest are 20- to 100-ppm range; must meet DQOs.	Initial must bracket routine sample concentrations.	Continuing checks to bracket the analytical run. Minimum of 1 blank/day.	±30%	Calibration standards must be identified and well distributed over the calibration range; selected based on DQO. Recommend duplicates run at a frequency of 1 per 20 samples. DQOs provide additional QC.
				Minimum of 1 blank/day.	<3 X MDL	
Indicator kits (colormetric) immunoassay	Field measurement for a variety of analytes.	Very low ppm range; must meet the DQOs.	Initial by manufacturer.	Continuing checks to bracket the analytical run.	±30%	Check expiration dates and storage requirements. Recommend duplicates at a frequency of 1 per 20 samples. DQOs may require additional QC.
				Minimum of 1 blank/day.	<3 X MDL	
Alpha spectroscopy	Determines the identity and content of alpha radionuclides.	Project-specific; must meet DQOs.	Initial and after repair, major maintenance or if system control cannot be re-established.	1 background/monthly.	Represents background for the time when the sample is counted.	Project-specific calibration may apply. DQOs may require additional QC.
				Daily instrument verification.	+20% of calculated activity.	
Gamma spectrometry	Determines the identity and curie content of gamma-emitting radionuclides.	Project-specific; must meet DQOs.	Initial and after repair or major maintenance or if system control cannot be re-established.	1 background/monthly.	Represents background for the time when the sample is counted.	Project-specific calibration may apply. DQOs may require additional QC.
				Daily source verification.	±10% of the source activity.	

**Table 5.2. Field Testing Instrumentation Quality Requirements.**

<b>Instrument</b>	<b>Use</b>	<b>Detection Limits</b>	<b>Calibration</b>	<b>QC Requirements</b>	<b>QC Criteria</b>	<b>Comments</b>
Liquid scintillation	Analysis of alpha and beta activity, including radionuclides (useful for low-energy radionuclides).	Project-specific; must meet DQOs.	Initial and after repair, major maintenance, or if system control cannot be re-established.	Quench curves for specific cocktail.	±20% of calculated activity.	Project-specific calibration may apply. DQOs may require additional QC.
				One laboratory control sample per 20 samples of the same matrix.	±20% of calculated activity.	
Gross alpha counting	Analysis of total alpha emitters.	Project-specific; must meet DQOs.	Initial and after repair, major maintenance, or if system control cannot be re-established.	One background/ monthly.	Represents background for the time when the sample is counted.	Project-specific calibration may apply. DQOs may require additional QC.
				Daily instrument verification	Three sigmas	
				One laboratory control sample per 20 samples of the same matrix.	±20% of calculated activity.	
Beta counting	Analysis of total beta emitters.	Project-specific; must meet DQOs.	Initial and after repair or major maintenance or if system control cannot be re-established.	One background/ monthly.	Represents background for the time when the sample is counted.	Project-specific calibration may apply. DQOs may require additional QC.
				Daily instrument verification	Three sigma.	
				One laboratory control sample per 20 samples of the same matrix.	±20% of calculated activity.	

Performance check standards are analyzed in accordance with the instrument-specific criteria as stated in instrument-specific procedures or manufacturer's criteria. Performance check standards shall meet the QC specified in the instrument-specific procedure before analysis can begin or continue.

### **5.1.3 Duplicate Analysis**

Duplicate analysis consists of performing analyses on two samples of the same population carried through all steps of the analytical method. For field-testing methods, a duplicate analysis consists of one sample per batch of 20 or fewer samples, or as otherwise specified in the approved sampling document. Depending on the analytical methods, this may be a sample duplicate or a matrix spike duplicate. Duplicates for some radiological testing methods may consist of recounting samples with the same instrument in the same geometry and comparing the results. Duplicates are used to assess the precision of the preparation and analytical/counting process in a client-specific matrix. Agreement between duplicates indicates the reproducibility (i.e., precision) of the measurement process. Disagreement can occur due to analyte concentration differences within the sample matrix (e.g., non-homogeneity) that are not amenable to analyst control during the analytical process (e.g., isolated particles of analyte in a soil matrix that cannot be reliably sampled using standard laboratory subsampling techniques). Disagreement may arise if the method has poor applicability to the analyte/matrix system.

For field screening methods, duplicate analysis consists of replicate measurements of the same property under prescribed similar conditions and is performed if specified in the field screening procedure or required by the client. Replicate measurements evaluate instrument operator precision and instrument precision. Disagreement for radiological measurements can occur when there is radioactive decay of very short-lived nuclides, sample self-absorption, sample thickness variances, and other radiological interferences.

### **5.1.4 Matrix Spike**

In general, a matrix spike is a client sample that has been spiked with the analyte(s) of interest and is processed in the same manner as the sample. The matrix spike is used to monitor method performance in a specific sample matrix. Matrix spike results are indicators of the effect that the client sample matrix has on the accuracy of measurement of the target analytes. If required by the method, one spike sample per batch shall be analyzed. Matrix spikes are not commonly performed for radiological field analytical methods in a field environment. If a client or method requires matrix spike samples, then Volume 4, Section 6.0 should be consulted for relevant requirements.

Each field analytical organization shall evaluate matrix spike recovery information, if applicable, against client data quality requirements. The goal is to ensure that limitations on the data caused by the sample matrix and represented by matrix spike performance are adequately portrayed and discussed in the report to the client.

### **5.1.5 Instrument-Specific Quality Control**

Most of the analytical methods used in the field have instrument-specific QC requirements. Tables 5.1 and 5.2 include additional detailed information. In all cases, the analyst shall demonstrate that the method is in control prior to performing sample analysis. If the instrument-specific QC requirements are not met before or during analysis, the analysis shall be terminated and the source of the problem identified and corrected.

## **5.2 GENERAL FIELD ANALYTICAL QUALITY CONTROL**

In this section, QC requirements for collection and analysis of samples using radiological, inorganic, and organic analytical procedures are described. The use of non-certified reagents and standards, materials, and equipment and methods of lesser quality can result in added interferences, smaller precision and less accuracy. These conditions are likely to be encountered in field applications. Each field analytical group shall have a mechanism in place for demonstrating control over such sources. A list of sources is described in the following subsections.

### **5.2.1 Distilled or Deionized Water**

High-purity water is generally defined as water that has been distilled or deionized (or both) so it will have conductivity less than 1.0 mho/cm (greater than 1.0 megaohm-cm resistivity). Each field analytical group shall ensure that water used for data collection activities is of sufficient quality for the operation performed. Water quality shall be regularly monitored through analysis of method blanks when method blanks are required.

### **5.2.2 Compressed Gases**

Each field analytical group shall monitor the quality of gases used in the field instruments to ensure that they are adequate for the operation being performed. At a minimum, this shall consist of monitoring system performance (e.g., for contribution to background and/or blanks from impurities).

### **5.2.3 Reagents**

Each field analytical group shall use reagents for data collection activities that are of sufficient quality for the operation performed. Reagent quality shall be regularly monitored by preparative and analytical QC performance.



#### **5.2.4 Lab-ware**

Each field analytical group shall purchase and use lab-ware of sufficient quality to meet client requirements. Lab-ware selected shall be compatible with the testing performed.

#### **5.2.5 Glassware Cleaning**

Glassware cleaning shall be performed in a manner that minimizes sample contamination.

#### **5.2.6 Good Housekeeping**

Each field analytical group shall maintain operations in a clean and organized manner to maximize available workspace and minimize environmental impact to sample quality. For radiological testing and field screening activities, the workspace must include radiological controls to reduce the potential for increase background activity from other field or laboratory activities, the presence of other samples near the counting equipment, and the potential detector contamination from other samples.

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## **6.0 DATA COLLECTION, REDUCTION, REVIEW, AND REPORTING**

Data collection and reporting processes include proper sampling, accurate chain of custody, proper collection of raw data, accurate data reduction and calculation, and the precise transfer of results to a final form. After the data are collected, reduced, and reviewed, the data are reported to the client in an easy-to-use form. A copy of the report to the client and all supporting analytical information used to generate the report are then assembled and archived as part of the permanent project record. This section discusses how these processes are applied to field screening and field-testing methods.

All of the records described in this section shall be maintained in a traceable manner as part of the permanent project file to safeguard the data and meet regulatory requirements as described in Volume 1, Section 6.0. This record keeping process will permit the reconstruction of all relevant activities that were used to produce the reported data.

### **6.1 DATA COLLECTION**

Raw data include all parameters used to calculate a final reportable result. Raw data can be generated by manual and/or electronic means. Manual data-generation records shall be collected and documented according to applicable procedures. Field screening and field testing frequently involve the use of field logbooks and forms for real-time data collection. Some field screening and field-testing instruments may electronically record field measurement information that is later downloaded or otherwise transferred to more-permanent forms of storage. Field analysis performed at an onsite facility frequently captures manually generated data in logbooks, forms, or by equivalent means, or as electronic records that are not interfaced to central data storage devices used in fixed laboratory applications. Procedures shall be in place that detail how these field records are captured and protected as part of the permanent project file.

Many instruments are interfaced with computers and/or integrators and are capable of generating and/or reducing raw data into reportable results. Procedures or written instructions shall be in place to describe the use of automated instruments and will address the processing of data for reporting to the client and the use of instrument-generated reports that are transmitted to the client. To the extent practical, both forms of data (i.e., manually calculated and generated by instruments) should be reported to the client in the same format, with the same number of significant figures when the two sources of data are used to meet the same information requirement as specified in the DQOs, SAPs, or other planning documents.

Entries into logbooks, forms, or equivalent shall be made in a manner that can be easily read, understood, and reproduced with a standard photocopier. Indelible ink shall be used. Corrections shall be made by drawing a single line through the erroneous entry and then initialing and dating the correction.

Data output shall be retained as a part of the project records (see Volume 1, Section 6.0). Information on the dates of field screening, sample collection, sample preparation, and field testing; sample identification numbers; analyst or instrument operator; type of analysis; and procedure number (including the revision number) shall be traceable to the raw data output.

## 6.2 DATA REDUCTION

Data reduction is defined as the mathematical operations that are applied to raw data to produce a final reportable result. Data resulting from analyzing samples shall be reduced according to applicable procedures. Data reduction includes activities that convert instrument and computer responses into reportable results. These activities may involve calculations, changes to the units or data values, and statistical and mathematical analysis.

The following practices shall be established in the field analytical organization's data review and/or verification procedures or QA plan to ensure accuracy of data entry, proper calculation, and appropriate data reduction:

- Verify that all readings/outputs are accurate.
- Ensure proper error correction or data change (i.e., one line through, dated, initialed, and explained, as appropriate).
- Select appropriate formulas for calculating final results, to correct for background and/or interference (e.g., Compton effects for gamma energy analysis and inter-element correction for inductively coupled plasma), and to document calculations and results. These formulas shall be included as an integral part of each method's standard operating procedure.
- Verify that data are accurately transcribed into notebooks, forms/benchesheets, spreadsheets, or other electronic formats (e.g., databases).

### 6.2.1 Significant Figures

Significant figures reflect the limits of the particular analysis method. Basic rules for significant figures and for calculating values and retaining the number of significant figures are provided in *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications* (ASTM 1999). Reported values should contain only significant figures. The number of significant figures should not exceed data needs, as specified in project DQOs, SAPs, or other planning documents.

Recognizing that vendor-supplied software may not meet the general rules for significant figures, the field analytical organization should work with the client to determine the best way to report results based on the project's needs.

### 6.2.2 Rounding-Off Methods

When a figure is to be rounded to fewer digits than the total number available, the rounding-off procedure described in ASTM (1999) should be followed. If a different rounding method is used, the method shall be noted in the narrative. A brief description of the procedure follows.

- When the first digit discarded is less than five, the last digit retained should not be changed.
- When the first digit discarded is greater than five, the last figure retained should be increased by one.
- When the first digit discarded is exactly five followed only by zeros, the last digit retained should be rounded upward if it is an odd number, but no adjustment made if it is an even number.

Recognizing that vendor-supplied software may not meet the general rules for rounding-off figures, the field analytical organization should work with the client to determine the best way to report results based on the project's needs.

### 6.2.3 Data Verification and Review

Data verification is the process used by the analytical service organization to confirm that all work requested by the client has been performed properly. The SAP (FSP and QAPjP), client communications, chain-of-custody forms, and other appropriate documents establish the specific information and quality requirements for each project. Verification is performed in accordance with these requirements using the information contained in the analytical report(s) to the client and all supporting documentation generated and retained by the analytical organization. The analytical service organization should establish a procedure to conduct this verification.

Data review refers to the process of determining whether data conform to requirements specified in the project DQOs, SAPs, or other planning documents. All individuals involved in generating the data are responsible for the quality of the work in the final report. Initial data review is conducted by the analyst during data collection. Individuals involved in steps such as data entry, data transcription, reduction, and project file assembly should review their work product before forwarding it to the next stage in the reporting process. If questions arise about the work product from preceding steps in the process, steps should be taken to resolve the questions before forwarding the work product to the next stage.

All data reported to the client should be technically reviewed by an individual other than the individual(s) who generated the results. Analysis performed by a field analytical organization should receive independent review prior to being released to the project staff. In applications, where data are being used in real-time to support project activities (e.g., most often field screening supporting site remediation, screening for areas of elevated contamination, etc), procedures that describe the types of real-time calibration confirmation and/or QC performance shall be in place, and the field analyst must document. The QC performance documentation shall be in place before providing the data directly to project personnel in real-time. If the data

generation is sufficiently complex or the intended use is critical to the project (e.g., pass/fail regulatory criteria or health and safety), it is recommended that a second qualified individual be available to conduct an independent review within the timeframe needed by the project.

All review and reporting procedures shall be described in project documents or field analytical organization's standard operating procedures. In the event that data must be reported without a prescribed review (e.g., in the case of emergency turnaround or inability to conduct timely review of real-time data within 24 hours of reporting), the data shall be marked as "DRAFT" or "PRELIMINARY" to indicate that the necessary reviews have not been completed according to procedure. In the event the data will not receive a data review, the data should be marked "UNREVIEWED DATA." Errors discovered in real-time data that have been reported to the client shall be reported to the client immediately upon discovery, with an estimate of the type of error that occurred. The errors shall then be referred to the analyst or other appropriate individual for corrective action (see Volume 1, Section 5.0). Timely notification of project personnel is critical to prevent potentially hazardous or regulatory noncompliant decisions to be made while the field analytical organization carries out the necessary corrective action.

The data review process shall incorporate the following elements.

- Data shall be reviewed according to the analytical organization's approved procedures to verify that calculations are correct and to detect transcription errors.
- Data shall be reviewed against applicable criteria for calibration, continuing calibration verification, QC, and other method criteria as appropriate to verify that the preparative and/or analytical system is performing acceptably (see Section 5.0 of this volume for further details). If QC samples do not meet established criteria, data within the batch shall be evaluated to determine if there were any adverse effects with respect to the client's requirements. If applicable, the sample(s) shall be re-prepared and/or re-run, or the data shall be reported with an explanation and appropriate qualification(s), which will be detailed in the narrative.
- Random checks shall be performed to verify calibration, data entry, calculations, and QC criteria.

### **6.3 DATA REPORTING**

The analytical information reported shall include the measured parameters, the details of analysis, and the reported data values. If applicable, the method procedure will provide data qualifiers and will note any QC failures.

Nonradiological results shall be reported as numeric values if the results are above the MDL. If the value is less than the MDL, it can be reported as undetectable.

Radiological measurement results should be reported based on calculated activity values (whether negative, positive, or zero) using the appropriate blank. The measured activity should

be reported with estimates of total propagated uncertainty, but without comparison to the estimated a priori minimum detectable activity (MDA) or minimum detectable concentration (MDC). The MDA or MDC should not be reported to the client in lieu of low-level measurements.

### **6.3.1 Data Reporting Documentation**

The reporting requirements for analysis shall be identified prior to field screening or field testing. The contents of the reports and the unreported information, retained as part of the permanent record, must demonstrate that the data meet the quality requirements specified in the planning documents, or the reasons that the requirements could not be met must be documented.

**6.3.1.1 Report Requirements for Field Screening and Field Testing.** Project DQOs/requirements frequently identify activities that are driven by real-time data acquisition, reporting, and decision-making. Examples of this are field screening or field-testing activities to identify areas of elevated contamination requiring remediation and providing data to support waste characterization and site characterization. These types of data needs are normally performed at Hanford using field screening and field testing. The minimum report requirements for this type of data are as follows:

- Field analytical organization name
- Project name.
- Unique sample identifier: This applies to field screening or field analysis where the field analyst performs the field screening or collects the sample(s) and later performs the field analysis without the sample leaving their possession. See the additional requirements listed in Section 6.3.1.2 for samples collected by a third party and relinquished to the analyst in the field or fixed laboratory.
- Analytical results and date of analysis (for each sample) with units (reported with an appropriate number of significant figures) and associated uncertainty, where appropriate.
- Detection limits or screening levels, as appropriate.
- Method references
- Statement that all calibration and QC criteria were met for the reported results
- Signature of person accepting responsibility for the report contents
- Date of issue

### **6.3.1.2 Report Requirements for Field Analysis for Regulatory or Compliance Activities.**

In addition to the information requirements listed in Section 6.3.1.1, the following information items are required to support activities associated with regulatory drivers such as waste characterization, waste shipment, permits, and site closure:

- Chain-of-custody form, including unique site identification, name, date and time of sample collection, unique client sample number, name of sampler(s), and names of people who accepted custody of the sample prior to arrival at the field laboratory or fixed laboratory. Note that information may be documented by providing a copy of the chain-of-custody form that is completed in the field.
- Sample information including unique laboratory identifier(if used), which is cross-referenced to client identification number, and date of sample receipt. Note that this information may be provided by providing a copy of the chain-of-custody form and associated sample receipt records after completion by the sample receiver.
- Date(s) of sample preparation, if applicable.
- Identification of any amended test results, if applicable.
- Identification of subcontracted results, if applicable
- Appropriate QC results (correlation with sample batch shall be traceable and documented)
- Appropriate data qualifiers with definitions and a narrative on the quality of the results, if applicable
- Additional data reporting (e.g., the percent moisture/solid or correction for equivalent dry weight), as appropriate

### **6.3.2 Emergency Reporting**

An immediate data reporting system shall be established between the field analytical organization and the client to address emergency turnaround situations. The type of information, level of approval, data reporting format, and means of delivery shall be discussed and agreed upon between the field analytical organization and the client.

## **6.4 COMMON DATA QUALITY CALCULATIONS**

This section provides various formulas that are typically employed to compute QC parameters for assessing data quality. These QC parameters shall be monitored, evaluated, and/or trended on a short-term and long-term basis, as needed. For example, system contamination control (blank or background activity), precision, accuracy, and spike recovery could be evaluated based on method, matrix, and activity or concentration level. Such activities provide a basis for



continuous quality improvement and insight regarding overall field analytical organization performance.

#### 6.4.1 Precision

Precision represents a measure of the degree of reproducibility of measurements under prescribed similar conditions. Sample precision is typically calculated on the basis of duplicate analyses or replicate measurements. Acceptance criteria shall be established for selected analytes and field screening or field-testing methods and shall be agreed upon by the field analytical organization and the client. Samples used to calculate precision should contain concentrations of the analytes of interest above the MDC or estimated quantification limit (EQL). The precision of a method in a given matrix is expressed as the relative standard deviation (RSD) or the relative percent difference (RPD).

In addition to precision determined by the sample duplicate or matrix spike duplicate, precision for the standards (e.g., laboratory control sample or continuing calibration verification standard) can be calculated and used to monitor QC of the analytical measurement system over time. Precision of the sample can also be monitored for long-term QC but should be based on method, matrix, and activity/concentration in the sample.

**6.4.1.1 Relative Standard Deviation:** The RSD is used when at least three replicate measurements are performed on a given technique. The RSD is computed using the following equation:

$$\text{RSD} = \frac{s}{\bar{x}} * 100$$

where

s	=	standard deviation with n - 1 degrees of freedom
n	=	total number of observed values
$\bar{x}$	=	mean of observed values

**6.4.1.2 Relative Percent Difference:** The RPD is used when two measurements exist. The RPD is generally used to express the precision of duplicate or spike duplicate samples and is computed using the following equation:

$$\text{RPD} = \frac{|x_1 - x_2|}{\bar{x}} * 100$$

where

$x_{1,2}$	=	observed values
$\bar{x}$	=	mean of observed values

## 6.4.2 Accuracy

Accuracy represents the degree to which a measurement agrees with an accepted reference or true value. Sample accuracy is typically expressed as the percent recovery of an analyte in a reference material or a spiked sample. Acceptance criteria shall be established for selected analytes and field analytical methods and shall be agreed upon by the field analytical organization and the client. Split samples may be submitted to a fixed laboratory for confirmatory quantification.

For radiological field screening and field-testing, accuracy is typically the variation of the measurements about some calculated mean, as determined by a succession of measurements of the same sample or reference. It should be noted that some variation in accuracy for radiological measurements is based on non-uniformity in decay rates over relatively short periods of time.

**6.4.2.1 Method Accuracy Based on Sample Spike:** Accuracy for the sample is expressed as the percent recovery (%R) of a matrix spike (or matrix spike duplicate) sample. The percent recovery is calculated based on the following equation:

$$\% R = \frac{(SSR - SR)}{SA} * 100$$

where

SSR = spiked sample result  
SR = sample result  
SA = spike added

**6.4.2.2 Method Accuracy Based on Standard:** The accuracy of an analytical method is expressed as the percent recovery of a standard (%R). The percent recovery of a standard is calculated by the following equation:

$$\% R = \frac{A_m}{A_k} * 100$$

where

$A_m$  = measured value of the standard analyte  
 $A_k$  = known value of the standard analyte

Method accuracy obtained from either, a sample spike or from a standard can be used to monitor QC of the analytical measurement system over time. Sample accuracy should be tracked based on the method, matrix, and activity/concentration when it is used for long-term QC monitoring.

### 6.4.3 Detection Limit Considerations

Detection limit determinations are performed to obtain information regarding the reliability of low-level results reported. A variety of approaches may be used, each of which portrays method sensitivity at low concentrations differently. The following subsections describe several typical detection limit determinations. Each field analytical organization shall document which approach is employed and describe how the determination is applied (e.g., performed in sample matrix or performed using low-level standards).

#### 6.4.3.1 Inorganic and Organic Methods

**6.4.3.1.1 Method Detection Limit:** The MDL is defined as “the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is greater than zero” (SW-846, consistent with the requirements specified in Appendix B to *40 Code of Federal Regulations* [CFR] 136) and is briefly described in the following text.

The concentration of the MDL for the analyte of concern can be estimated by using one of the following:

- An instrument signal-to-noise ratio within the range of 2.5 to 5
- The region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

When determining the MDL, a minimum of three analyses are required in a matrix spiking with the analyte of interest at a concentration three to five times the estimated MDL. Whenever possible, the matrix should be the same as or very similar to the sample matrix. All sample processing steps of the analytical method shall be included in the final determination of the MDL.

Variance ( $S^2$ ) is determined from the replicate measurements, as shown:

$$S^2 = \frac{\sum (X_i - \bar{X})^2}{(n - 1)}$$

where

$\frac{X_i}{\bar{X}}$  = with measurement of the variable X  
= mean of observed variable X.

The MDL should be determined by the following equation:

$$\text{MDL} = t_{(n-1, \alpha=.99)} * (s)$$

where

$t_{(n-1, \alpha=.99)}$  = one-sided t-statistical value appropriate for the number of samples used to determine standard deviation

s = standard deviation obtained from the MDL replicate measurements.

Each field analytical organization shall document its approach when employing the MDL determination. The term MDL shall only be employed when all method steps (sample preparation through analysis) are tested. The field analytical organization shall also document whether the determination was performed using client samples or standards. Results that are below the MDL should be reported as not detected.

**6.4.3.1.2 Instrument Detection Limit:** The IDL is determined by spiking reagent water with each analyte of concern. The following considerations apply to the selection of the IDL standard:

- Concentration of the IDL standard should be at least equal to, or in the same concentration range as, the estimated IDL.
- Concentration of the IDL standard should be in the region of the standard curve where there is significant change in sensitivity.

A minimum of seven aliquots of the IDL standard is required to determine the IDL. The IDL standards are run through the analytical process only. The IDL is calculated in the same manner as the MDL. In cases such as some organic analysis and mercury and cyanide determinations, the IDL standard should be subjected to preliminary extraction, digestion, and/or distillation. Results that are below the IDL should be reported as not detected.

**6.4.3.1.3 Practical/Estimated Quantitation Limit or Reporting Limit:** The EQL is the lowest concentration of an analyte that can be reliably achieved within specified limits of precision and accuracy during routine field analytical organization operating conditions. The analyte concentration at the estimated quantitation limit is determined using the following guidance: (1) 5 to 10 X MDL or IDL, or (2) the lowest non-zero standard in the calibration curve.

Each field analytical organization shall document the approach that is used to determine the EQL. The EQLs reported with client data shall reflect all method dilution factors (i.e., dilution factors resulting from sample preparation).

Results that are between the MDL and the EQL should be reported with appropriate qualification (e.g., flag or footnote).

### 6.4.3.2 Radiological Methods

**6.4.3.2.1 Decision-Level Count Rate.** The decision-level count rate (DLR) is defined as a 95% confidence limit for a critical decision level. This level is used for making the decision whether a sample emits radiation above the appropriate blank background level. The decision should be based solely upon whether the net count rate observed for that sample exceeds this DLR. The DLR is calculated as shown below:

$$DLR = 1.65 * \sqrt{\frac{R_b}{T_b} + \frac{R_s}{T}}$$

where

$R_b$	=	background count rate
$R_s$	=	sample count rate
$T$	=	sample count time
$T_b$	=	background count time.

When counting a sample that does not contain an analyte (i.e., radionuclide) of interest,  $R_s$  is assumed to be equal to  $R_b$ . The DLR can be simplified as shown below:

$$DLR = 1.65 * (S_b) * \sqrt{2}$$

where

$S_b$	=	standard deviation of background (or appropriate blank) count rate for the counting time (T).
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For the purpose of interpreting whether an individual sample measurement is different from its appropriate blank, it is recommended that the field analytical organization compare the net sample count rate with a decision level count rate calculated using the sample specific “appropriate” blank. The “appropriate” blank should include measurement interferences from impurities (e.g., elevated Compton continuum or channel cross-talk from higher energy alpha particles measured by liquid scintillation) that are not typically known *a priori* or included in the nominal *a priori* DLR limit. This “true” decision level for the sample is different from the nominal *a priori* decision limit. For some measurement processes, the determination of the “true” appropriate blank for each sample may be impractical. However, every effort should be taken to properly assess the parameters of the appropriate blank.

**6.4.3.2.2 Minimum Detectable Activity:** The MDA has been defined as a level of activity that is practically achievable by a measurement system. The sample MDA generally is applied as the mean (expected) activity of samples having a 5% probability of escaping detection and 5% probability of false detection. The MDA is calculated based on Currie's (1968) formula and is simplified to the following two equations when the counting time in the sample is the same as in the background:

$$MDA = \left[ \left( \frac{2.71}{T} \right) + (2 * DLR) \right] / K$$

or

$$MDA = \left[ \left( \frac{2.71}{T} \right) + (4.65 * S_b) \right] / K$$

where

- T = sample count time
- K = detector calibration factor (e.g., count rate/disintegration rate)
- S<sub>b</sub> = standard deviation of background count rate for the counting time (T).

When T<sub>b</sub> is not equal to T<sub>t</sub>, the MDA is calculated as shown below:

$$MDA = \frac{2.71 + 3.3 \sqrt{(R_b * T_b) * \left( 1 + \frac{T_b}{T_t} \right)}}{\xi * b * T_t * k}$$

where

- R<sub>b</sub> = background count rate
- T<sub>b</sub> = background count time
- T<sub>t</sub> = sample count time
- ξ = counting efficiency
- b = abundance
- k = conversion factor to convert to desired units

The MDC is defined as the mean concentration of samples having a 5% probability of escaping detection and 5% probability of false detection.

$$MDC = \frac{MDA}{q * \text{decay}}$$

where

q = sample quantity (e.g., g or ml)  
decay = decay factor (correction for radioactive decay to reference date).

Software provided by vendors may use variations of the above formula. Vendor-provided software or data reduction packages are adequate for data calculation.

**6.4.3.2.3 *A Priori* and *A Posteriori* Concepts and Information:** The DLR, MDA, and MDC are considered to be *a priori* (before the measurement). The estimation of these quantities requires specification of nominal values of a number of parameters (e.g., background count rate, count time, estimated interferences, chemical recoveries, and decay times). The true appropriate blank for a measurement process includes estimates of the nominal levels of any interference that may be present in a sample batch. In a number of situations, regulatory limits or contract specifications may require that the measurement process meet or exceed certain MDC limits for the sample batch of interest. Because these determinations may require preliminary measurements, the assessment of *a priori* detection limit parameters for future measurements may require the knowledge of *a posteriori* information regarding the nominal characteristics of the sample batch gained from preliminary measurements.

#### 6.4.4 Uncertainty

Uncertainty is expressed as the range of values where the true value is estimated to lie. The uncertainty estimate consists of two components: systematic and random variability. Each contributing source of uncertainty is expected to be distributed over the range. Each systematic component can be estimated in terms of the measurement result for the contributing source of uncertainty. The analytical systematic component can be estimated using standard or spike recovery. The random analytical component can be estimated from replicate measurements of a sample. The total uncertainty is calculated as the square root of the sum of the squares of random and systematic variabilities as shown in the following equation. The component of uncertainty has to be expressed in the same unit designation (e.g., concentration percentage).

$$\text{Total uncertainty} = \sqrt{(s_x^2) + \sum_{j=1}^q \delta_j^2}$$

where:

$s_x$  = standard error  
q = number of systematic uncertainty component  
 $\delta$  = systematic uncertainties.

Uncertainty is commonly used in the radiochemical analyses to express method and counting error. The total random uncertainty is obtained by propagating the individual variance ( $s_i^2$ ) and is expressed as the standard error based on multiple determinations of  $x$ . However, the typical radiochemical methods used are not sufficient to separate systematic and random uncertainties such that biases can be corrected. Uncertainty will be measured, or uncertainty will be estimated if it cannot be measured.

#### **6.4.5 Control Charts**

Control charts provide the analyst with early warning of impending problems in a preparative or analytical method and make bias trends more obvious. Each field analytical organization shall document its policy regarding the use of control charts. The organizational policy shall articulate the manner in which the organization will deal with statistical outliers. Blank spike/laboratory control sample performance for all routine preparations shall be monitored using control charts. In cases where the analytical technique involves a large number of analytes (e.g., inductively coupled plasma or gas chromatography/mass spectrometry), the field analytical organization may select a subset representative of the total for control charting. Performance statistics can be tabulated in lieu of a control chart.

Control charts are useful for monitoring the performance of field radiological instruments and should be used to help control field measurement systems. A separate control chart for calibration checks, background measurements, and source checks should be developed for each instrument.



## **7.0 DATA VALIDATION AND ASSESSMENT**

This section provides guidance to the field analytical organization in the areas of data validation and assessment that may be required by projects. While the field organization is not directly responsible for validation or assessment, the organization plays an important supporting role.

Data validation is the process where the client or project may submit the analytical data (including appropriate supporting documents provided by the field analytical organization) to a qualified, independent third party for review. The third party compares the information with the project DQO and other planning documents, the field analytical organization QA/QC requirements, and prevailing industry or regulatory standards. This comparison results in a validation report that identifies any quality gaps between the client's expectations (DQOs and planning documents), field analytical organization QA/QC program, industry standards, the analytical data, and supporting information. The analytical service organization has no role in the validation process beyond the timely submission of copies of all information requested by the third party organization. The analytical service organization should be available to provide any additional information requested by the third party as the process proceeds. The service organization should establish a procedure to provide and track the information submitted to the client (or project) and the third-party organization.

Data quality assessment is the process used by the client or project to examine the analytical data, sampling data, and other appropriate information to determine if the data are adequate for the intended use. The data quality assessment may be included in verification and validation or may be performed independently.

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