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Plan

QUALITY ASSURANCE PROJECT PLAN FOR ANALYTICAL LABORATORIES DEPARTMENT RADIOANALYTICAL ACTIVITIES

Idaho National Engineering
and Environmental Laboratory

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ABSTRACT

This Quality Assurance Project Plan specifies quality assurance/quality control procedures employed by the Analytical Laboratories Department in the performance of radioanalytical activities. It provides radioanalytical-specific implementation of Company quality program requirements, as specified in PRD-5071, "Quality Assurance Program". This Quality Assurance Project Plan is written to conform with the requirements and guidelines specified in MCP-561, "Quality Program Plan/Quality Assurance Project Plan Development."

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ACRONYMS

ACLP	Analytical Chemistry Laboratory Procedure
ACMM	Analytical Chemistry Methods Manual
AEMP	Air Effluent Monitoring Program
ALD	Analytical Laboratories Department
Am	americium
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
ASPEP	Analytical Services Performance Evaluation Program
ASTM	American Society for Testing and Materials
Bq	Becquerel
C	carbon
CF	Central Facilities
CFA	Central Facilities Area
Co	cobalt
COC	chain of custody
cpm	counts per minute
Cs	cesium
DOE	U.S. Department of Energy
DOELAP	U.S. Department of Energy Laboratory Accreditation Program
dpm	disintegrations per minute
DRC	data and records coordinator
EA	Environmental Affairs
EES	Environmental and Energy Sciences Directorate
EO	Environmental Operations
EPA	U.S. Environmental Protection Agency
ESH &QA	Environmental, Safety, Health, and Quality Assurance
Eu	europium
GL	Group Lead
GS	gamma spectroscopy
GSTL	Gamma Spectroscopy technical leader
^{3}H	tritium
I	iodine
ICP	Idaho Completion Project
INEEL	Idaho National Engineering and Environmental Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
ISP	Interlaboratory Comparison Studies Program
IVA	In Vitro Analysis
IVATL	In Vitro Analysis technical leader
K	potassium
KeV	kilo-electron volts
Kr	krypton

LCS	laboratory control samples
LEPS	low energy photon spectrometry
LIMS	Laboratory Information Management System
M&TE	measurement and test equipment
MAPEP	Mixed Analyte Performance Evaluation Program
MCP	management control procedure
MDA	minimum detectable activity
MDC	minimum detectable concentration
MS	matrix spike
MSD	matrix spike duplicate
NE-ID	DOE Nuclear Energy, Science, and Technology Idaho Operations Office
Ni	nickel
NIM	National Instrument Module
NIST	National Institute of Standards and Technology
NPL	National Physical Laboratory
NQA	nuclear quality assurance
NRIP	NIST Radiochemistry Intercomparison Program
ORNL	Oak Ridge National Laboratory
pCi	picocurie
PdI_2	palladium iodide
PRD	Program Requirements Document
PTB	Physikalisch-Technische Bundesanstalt
PE	performance evaluation
Po	polonium
Pu	plutonium
QA	quality assurance
QAO	quality assurance officer
QAPjP	quality assurance project plan
QA/QC	quality assurance/quality control
QC	quality control
R&D	research and development
RA	radioanalytical
Ra	radium
RC	radiochemistry
RESL	Radiological and Environmental Sciences Laboratory
RML	Radiation Measurement Laboratory
RPD	relative percent difference
RSD	relative standard deviation
RTL	radiochemistry technical leader
SAM	Sample and Analysis Management
Sb	antimony
S&CL	Standards and Calibration Laboratory
SOW	statement of work

Sr	strontium
TL	technical leader
TRA	Test Reactor Area
U	uranium
VAXGAP	VAX System Gamma Spectrometry Analysis Programs
Y	yttrium

1. PROJECT DESCRIPTION

The Department of Energy (DOE) Nuclear Energy, Science, and Technology Idaho Operations Office (NE-ID) requires all laboratories supplying analytical services to develop, implement, and maintain a quality assurance project plan (QAPjP) that addresses all pertinent requirements of American Society of Mechanical Engineers (ASME) Nuclear Quality Assurance (NQA)-1 standard, as required by DOE Order 414.1A, "Quality Assurance". This QAPjP satisfies this requirement for the radioanalytical (RA) activities of the Idaho National Engineering and Environmental Laboratory (INEEL) Analytical Laboratories Department (ALD).

This QAPjP defines implementation of the INEEL quality assurance program requirements as specified in PRD-5071, "Quality Assurance Program" , specific to routine RA operations. The RA quality assurance requirements and procedures in this QAPjP incorporate guidance from American National Standards Institute (ANSI) N42.23, "American National Standard Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories" (ANSI 1996b) and American Society for Testing and Materials (ASTM) D3648-95, "Standard Practices for the Measurement of Radioactivity" (ASTM 1995). Requirements and practice for in vitro analyses defined in this QAPjP conform to the requirements of ANSI N13.30, "Performance Criteria for Radiobioassay" (ANSI 1996a).

This document complies with management control procedure (MCP)-561, "Quality Program Plan/Quality Assurance Project Plan Development." The format specified by MCP-561 is that used in EPA QA/R-5, EPA Requirements for Quality Assurance Projects (EPA 1999).

Table 1-1 provides a cross-reference between sections of this QAPjP and the basic requirements of ASME NQA-1.

For specific projects, the requirements in this document may be augmented or superseded by project-specific quality requirements documents (e.g., statements of work [SOWs], run plans, sampling and analysis plans).

The INEEL ALD RA laboratories perform radioanalyses for various customers and programs , including reactor and process operations, environmental monitoring, waste characterization, research and development (R&D), and personnel exposure monitoring. The RA laboratories are organized into three distinct operations; identified as "INTEC Radioanalytical," "Test Reactor Area (TRA) Radioanalytical," and "Central Facilities (CF) Analytical Chemistry." Each group includes Radiochemistry (RC), and Gamma Spectroscopy (GS) functions.

Table 1-1. Cross-reference between sections of this QAPjP, ASME NQA-1 criteria.

Section of Radioanalytical QAPjP	Basic Requirement of ASME NQA-1
1. Project Description	2. QA Program
2. Project Organization and Responsibility	1. Organization
	7. Control of Purchased Items and Services
	8. Identification and Control of Items
	9. Control of Processes
3. Quality Assurance Objectives	8. Identification and Control of Items
	9. Control of Processes
	10. Inspection
4. Sampling Procedures	—
5. Sample Custody	3. Design Control
	7. Control of Purchased Items and Services
	13. Handling, Storage, and Shipping
6. Calibration Procedures and Frequency	12. Control of Measuring and Test Equipment
	14. Inspection, Test, and Operating Status
7. Analytical Procedures	3. Design Control
	5. Instructions, Procedures, and Drawings
	6. Document Control
	9. Control of Processes
8. Data Reduction, Validation, and Reporting	3. Design Control
	6. Document Control
	9. Control of Processes
	17. QA Records

Table 1-1. (continued).

Section of Radioanalytical QAPjP	Basic Requirement of ASME NQA-1
9. Internal Quality Control Checks and Frequency	2. QA Program
	12. Control of Measuring and Test Equipment
	14. Inspection, Test, and Operating Status
10. Performance and System Audits and Frequency	2. QA Program
	15. Control of Nonconforming Items
	16. Corrective Action
	18. Audits
11. Preventive Maintenance	12. Control of Measuring and Test Equipment
12. Specific Routine Procedures for Quality Assurance	12. Control of Measuring and Test Equipment
13. Corrective Action	3. Design Control
	9. Control of Processes
	15. Control of Nonconforming Items
	16. Corrective Action
14. Quality Assurance Reports to Management	17. QA Records
15. Records Management	6. Document Control
	17. QA Records
16. Personnel Training and Qualification	9. Control of Processes
17. Procurement	4. Procurement Document Control
	7. Control of Purchased Items and Services
18. References	5. Instructions, Procedures, and Drawings

Radiochemistry laboratories are located at TRA and INTEC. Radiochemistry laboratories perform radiochemical analyses on environmental samples, such as soil, water and vegetation, as well as higher activity samples in support of characterization and R&D programs. These analyses include determination of alpha-emitting nuclides, such as plutonium (Pu)-238, uranium (U)-235, and americium (Am)-241; and beta-emitting nuclides, such as strontium (Sr)-90 and tritium (^{3}H).

The In Vitro analysis laboratories are located at CFA. The IVA laboratories perform radiochemical analyses on human waste products in support of Radiation Dosimetry Programs. These analyses include alpha-emitting nuclides, such as ^{238}Pu, ^{235}U, and ^{241}Am; beta-emitting nuclides, such as ^{90}Sr and ^{3}H; and gamma-emitting nuclides, such as cesium (Cs)-137 and potassium (K)-40.

Gamma spectrometry functions are located at TRA (the Radiation Measurements Laboratory [RML]), INTEC, and CFA. The GS laboratories perform analyses of gamma-emitting nuclides, such as cesium (^{137}Cs), cobalt (Co)-60, and antimony (Sb)-125, in support of Site Operations, Radiation Dosimetry, and Environmental Management, as well as other site-specific and R&D programs.

2. PROJECT ORGANIZATION AND RESPONSIBILITY

The ALD is part of the INEEL's Environmental and Energy Sciences organization. The relationship of the Environmental and Energy Sciences organization to other INEEL organizations is depicted in company organization charts maintained by the Human Resources Department and published on the INEEL intranet.

The functional organization of the ALD RA laboratories is shown in Figure 2-1. Roles and responsibilities within these laboratories are defined in the following subsections.

2.1 Analytical Laboratories Department Manager

The ALD manager is responsible for managing and overseeing laboratory operations and product quality for all ALD laboratories. The ALD manager's responsibilities include, but are not limited to, department policy development, quality assurance program implementation, resource allocation, work status and cost control monitoring, procurement approval, oversight of preventive and corrective action implementation, and negotiation of project requirements with customers. The ALD manager is responsible for ensuring that the laboratories within the department operate in compliance with company environmental, safety and health requirements. The ALD manager directs self-assessments of related ALD activities.

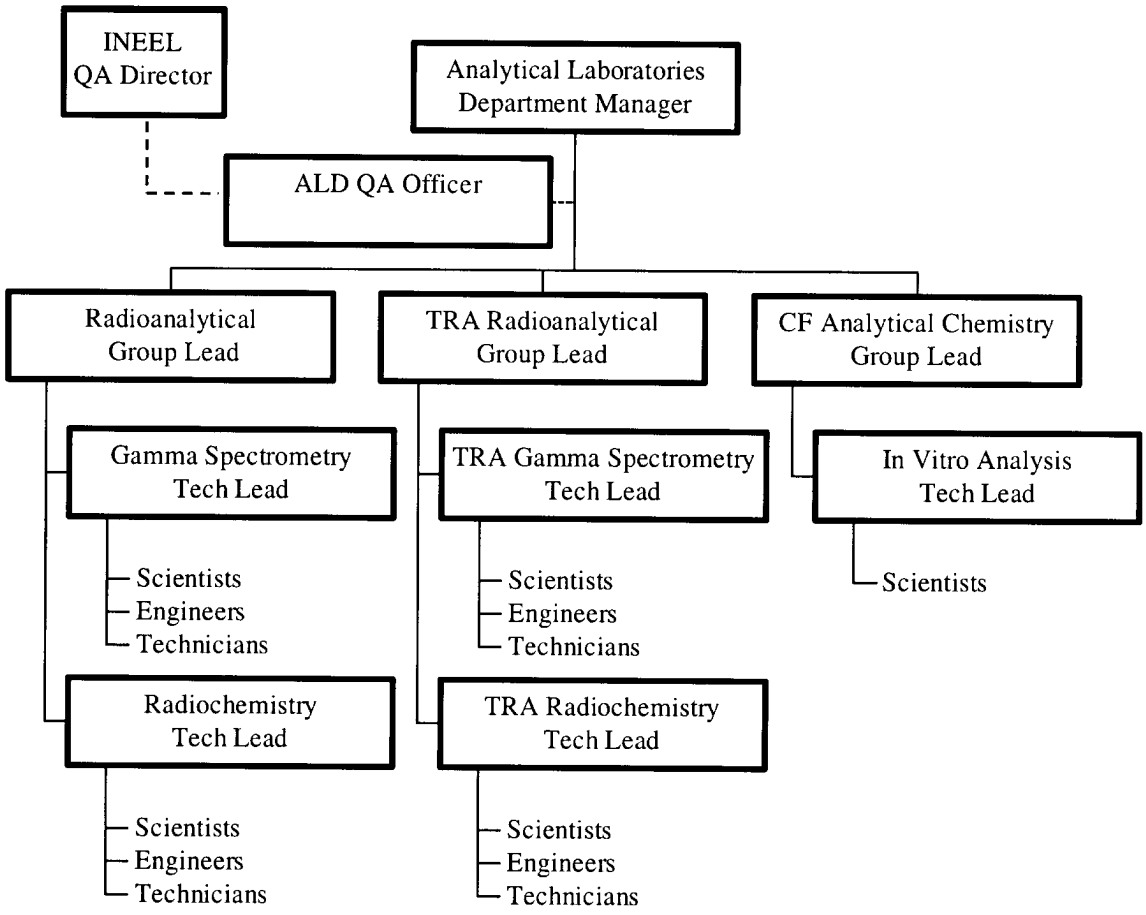

Figure 2-1. Functional organization of the Analytical Laboratories Department Radioanalytical Groups

2.2 Analytical Laboratories Department Quality Assurance Officer

The ALD quality assurance officer (QAO) is responsible for the oversight of all QA activities within the ALD. The ALD QAO reports directly to the ALD manager and is organizationally and functionally independent of RA production activities. The ALD QAO assists the ALD manager in developing department QA policy and provides independent oversight of QA policy implementation and practice. The ALD QAO provides QA training for ALD personnel. The ALD QAO is responsible for assisting ALD staff with developing, documenting, and evaluating quality assurance/quality control (QA/QC) procedures and practices. The ALD QAO works with cognizant ALD Group/Technical Leaders to resolve disputes related to RA QA requirements. If not resolved, the ALD QAO elevates the issue progressively to successively higher levels of management, as necessary for resolution. The ALD QAO coordinates corrective action tracking and implementation for the department and performs independent assessments of department operations. The ALD QAO reviews and approves all department QA plans and implementing procedures and periodically performs QA reviews of data. The ALD QAO is responsible for maintaining and revising this QAPjP.

The ALD QAO is not a quality engineer and does not fulfill quality engineer responsibilities as specified in company QA procedures (e.g. MCP-561); the ALD obtains quality engineering support from the company Quality Assurance organization when needed.

2.3 Radioanalytical Group Leads

The RA Group Leads (GL)s are responsible for overall coordination of the RA laboratories' activities. The GLs report directly to the ALD Manager and are responsible for ensuring that RA data are generated in accordance with this QAPjP and written and approved analytical procedures. The GLs appoint their respective staff members and ensure that they receive appropriate training. The GLs negotiate project requirements with customers, manage the implementation of project responsibilities, and control project costs. The GLs manage resource allocation within their respective laboratories and approve procurement actions per company policy. The GLs are responsible for drafting RA implementing procedures or for assigning those tasks to appropriate RA personnel. The GLs review and approve all RA implementing procedures.

2.4 Technical Leaders

Technical leaders (TLs) report to the GLs and are immediately responsible for direction of RA activities at the work level. The TLs are responsible for ensuring generation of technically valid data, coordinating and scheduling work, and training personnel. The TLs are responsible for ensuring that QA practices meet the requirements of this QAPjP and that quality control (QC) practices are implemented at required frequencies. The TLs are responsible for informing the ALD QAO and the respective GL of any concerns pertaining to data quality and for implementing corrective actions when required. The TLs provide technical direction for improving existing laboratory procedures and developing new ones. The TLs coordinate the data reporting processes and ensure that all RA data receive proper technical review prior to release to customers.

2.4.1 Radiochemistry Technical Leaders

The Radiochemistry technical leaders (RTL)s are responsible for directing the RC functions within their respective group. These functions include sample preparations, sample separations, and radiochemical determinations using alpha and beta spectrometry techniques. The RTLs coordinate with

the Gamma Spectrometry TLs (see section 2.4.3) to identify gamma spectrometry support requirements and needs for RC.

2.4.2 In Vitro Analysis Technical Leader

The In Vitro Analysis technical leader (IVATL) is responsible for directing the IVA functions. These functions include sample preparations, sample separations, and specialized radiochemistry techniques. The IVATL ensures that all approvals and certifications required for the laboratory's mission are obtained and maintained. The IVATL reviews and approves all In Vitro Analysis data packages and reports.

2.4.3 Gamma Spectrometry Technical Leaders

The Gamma Spectrometry technical leaders (GSTL)s are responsible for directing the GS functions within their respective group. The TRA GSTL is responsible for daily operation of the RML. The GSTLs coordinate with the RTLs and IVATL to ensure that all gamma spectroscopy measurements and analyses are performed within the guidelines of this QAPjP. The GSTLs provide technical guidance and direction to the GS personnel.

Scientists, Engineers, and Technicians

Laboratory scientists, engineers, and technicians are responsible for performing sample analysis, data reduction, and reporting in accordance with this QAPjP and program requirements. Scientists, engineers, and technicians are responsible for following analysis and QC procedures specified in analytical methods and documenting any deviation from methods or QAPjP specifications. Scientists, engineers, and technicians are responsible for critically observing and evaluating all analytical procedures and bringing any practices and occurrences that might affect the reliability of analytical data to the attention of the appropriate TL or GL, and the ALD QAO. They conduct the analyses, make computations, perform independent technical reviews, and transmit data to the appropriate TLs for review. Scientists, engineers, and technicians may be responsible for writing analytical methods at the direction of the TLs. Nuclear instrumentation systems support personnel are responsible for installing and servicing the nuclear instrumentation used by RC, GS, and IVA laboratories for counting samples.

2.6 Data and Records Coordinators

The data and records coordinators (DRCs) are responsible for maintaining the records as specified in this QAPjP. The DRCs work with the TLs to ensure that data reports meeting customer requirements are issued in a timely manner. The DRCs are responsible for maintaining laboratory analytical records, which include, but are not limited to, data analysis packages, QC results and summaries, and letter reports.

2.7 Sample Custodians

Sample custodians are the ALD personnel responsible for sample receipt and sample tracking at the laboratory. Sample custodians ensure that chain of custody (COC) is maintained when required (e.g., for environmental samples). After sample analysis and reporting is complete, sample custodians coordinate disposal of samples in accordance with company requirements.

2.8 Computer System Administrators

Computer system administrators are the ALD or matrixed personnel responsible for controlling and maintaining multi-user computer systems used at the INTEC and TRA laboratories. The computer system administrators control configuration of the multi-user computer systems and ensure that procedures are in place to prevent unauthorized changes to computer software and that all changes to the system are justified and documented. ALD MCP-2009, "Analytical Software Control," defines specific responsibilities for computer system administrators.

3. QUALITY ASSURANCE OBJECTIVES

Quality assurance objectives for RA laboratories address accuracy, precision, representativeness, completeness, and comparability. Minimum detection limits (minimum detectable activity) are also addressed.

3.1 Accuracy

Accuracy is a measure of how close a measured value is to a known true value. Accuracy is a also referred to as relative bias because it measures the bias of an analytical system. Accuracy is normally measured through the analysis of known standards (i.e., control samples) or use of radiotracers during analysis, and is expressed in terms of percent or fraction recovery or relative bias (see section 12.2). Accuracy quality assurance objectives for IVA (and GS support) are defined in ANSI Standard N13.30 (ANSI 1996a); relative bias must be between -0.25 and +0.50. The accuracy objectives for other RC and GS analyses are project specific and are defined in project-specific quality requirements documents (e.g., statements of work, run plans, sampling and analysis plans).

3.2 Precision

Precision is a measure of the ability to reproduce an analytical result, and it expresses the degree of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Various measures of precision exist depending upon the prescribed similar conditions. Precision is generally determined through the analysis of replicate samples (see definition) or by replicate analysis of a sample. Precision is best expressed in terms of standard deviation or relative percent difference (see section 12.2). Precision quality assurance objectives for IVA are defined in ANSI Standard N13.30. For IVA and supporting GS measurements, the relative precision (relative standard deviation) must be ≤ 0.40. The precision objectives for other RC and GS analyses are project specific and are defined in project-specific quality requirements documents (e.g., statements of work, run plans, sampling and analysis plans).

3.3 Representativeness

The representativeness objective expresses the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness cannot be quantitatively assessed. RA laboratories use standardized sample aliquotting and preparation and measurement techniques to ensure that analytical data produced are representative of the samples received at the laboratories.

3.4 Completeness

Completeness is a measure of the amount of valid analytical data obtained from a measurement system compared to the amount that was expected to be obtained under current normal conditions. Valid analytical data are those generated when analytical systems were in control, i.e., all calibration verification checks and other non-matrix checks (e.g., blanks and control samples) met project-specified acceptance criteria. The completeness objective for RA laboratories is normally 95%. Project-specific requirements may take precedent over this default value.

3.5 Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability cannot be quantitatively assessed. Comparability of data within the RA laboratories is ensured by use of standardized documented analytical methods and traceable standards. Due to a lack of national-consensus RA methods (e.g., Environmental Protection Agency [EPA] protocols), comparison with data sets generated by other laboratories is ensured through participation in performance evaluation (PE) programs sponsored by independent agencies and organizations. In addition, comparability of RA data within ALD is ensured by recording measurements in consistent units.

Performance evaluation program participation for the RA laboratories is summarized in Table 3-1. A brief description of each program and the RA laboratories' participation is presented in the following subsections.

Table 3-1. Radioanalytical laboratories' participation in performance evaluation programs.

PE Program	Sponsor	Required by	Program Participation by Section and Location					
				RC		GS		
			IVA	INTEC	TRA	INTEC	TRA	CFA
Mixed Analyte Performance Evaluation Program (MAPEP)	DOE-RESL	DOE-ID		X	X	X	X	
DOE Laboratory Accreditation Program	DOE-RESL	DOE (Certification)	X					X
ORNL Interlaboratory Comparison Studies Program (ISP)	DOE-ORNL	DOE (Certification)	X					X
NIST Radioanalytical Intercomparison Program (NRIP)	NIST	(voluntary)	X		X		X	X

ORNL = Oak Ridge National Laboratory
RESL = Radiological and Environmental Sciences Laboratory

3.5.1 Mixed Analyte Performance Evaluation Program

The Mixed Analyte Performance Evaluation Program (MAPEP) is administered by the DOE Radiological and Environmental Sciences Laboratory (RESL). Participation in this program is required by DOE-ID for all analytical laboratories performing environmental work. Samples containing radionuclides and metals are distributed semiannually, with sample matrices alternating between water and soil.

Radiochemistry and gamma spectroscopy laboratories at TRA (lab code LOCK03) and INTEC (lab code LOCK01) participate in the MAPEP for those analytes and matrices for which they perform routine analyses. The samples are analyzed using established ALD analytical methods. Results of MAPEP participation can be obtained from the MAPEP internet homepage (http://www.inel.gov/resl/mapep/) and are maintained on file at the ALD.

3.5.2 DOE Laboratory Accreditation Program

The DOE Laboratory Accreditation Program (DOELAP) is administered by DOE RESL for laboratories performing radiochemical analysis of In Vitro and In Vivo samples. Urine and fecal samples containing between 0.01 and 100 pCi of alpha, beta and gamma emitting radionuclides are provided once every three years. The IVA laboratory (and supporting GS laboratory) participates in this program as part of its DOE certification program. The samples are analyzed using established IVA analytical methods. The administrating organization provides a comparison of the results obtained by participating laboratories to the IVATL. Results of IVA DOELAP participation are maintained on file at the ALD.

3.5.3 ORNL Interlaboratory Comparison Studies Program

The Interlaboratory Comparison Studies Program (ISP) is administered by the Oak Ridge National Laboratory (ORNL) for laboratories performing radiochemical analysis of In Vitro samples. Urine and fecal samples containing between 0.01 and 100 pCi of alpha, beta and gamma emitting radionuclides are provided four times per year. The IVA laboratory (and supporting GS laboratory) participates in this program as part of its DOE certification program. The samples are analyzed using established IVA analytical methods. The administrating organization provides the results to the IVATL. Results of IVA ORNL ISP participation are maintained on file at the ALD.

3.5.4 NIST Radiochemistry Intercomparison Program

The National Institute of Standards and Technology (NIST) Radiochemistry Intercomparison Program (NRIP) is administered by NIST for laboratories performing radiochemical analyses. Samples are submitted annually, with sample matrices alternating between urine and fecal samples for In Vitro analyses, and between soil, water and air filters for environmental analyses. The samples contain between 2 and 20 pCi of alpha, beta, and gamma emitting radionuclides. The IVA laboratory (and supporting GS laboratory) and the RC and GS laboratories at TRA voluntarily participate in this program. Results of NRIP participation are maintained on file at the ALD.

3.5.5 Customer Performance Evaluation Programs

As part of their overall QA programs, RA customer organizations may conduct their own PE studies to verify the accuracy of their program data. These studies generally consist of single or double blind samples submitted to the laboratories along with actual program samples. Blind samples, prepared

by the customer, consist of commonly found radionuclides spiked onto water, soil, biota or air filters, as appropriate for the program. The RA laboratories analyze the PE samples concurrently with routine program samples and report results to the customer. Customers usually provide performance summaries to the GL and the ALD QAO.

Analysis results for a single customer-sponsored PE sample do not by themselves provide sufficient information to definitively evaluate laboratory performance. However, trends in customer-sponsored PE results, evaluated in conjunction with interlaboratory PE program performance and independent laboratory verification of PE material concentrations, are useful to identify measurement deficiencies and needed process improvements.

3.6 Minimum Detectable Activities

Minimum detectable activity (MDA) is an *a priori* estimate of the detection capabilities of a given measurement system and method for radionuclide analyses. This estimate is based on the premise that from knowledge of the background count and other measurement system parameters, an *a priori* limit can be estimated for a particular measurement. The MDA is defined on the basis of statistical hypothesis testing for the presence of activity. See section 12.2.4 for MDA calculation.

For laboratories performing environmental and characterization work, MDAs are specified in project-specific quality requirement documents, (e.g., statements of work). Minimum detectable activity quality assurance objectives for the IVA group (and supporting GS laboratory) are included in Table 3-2.

Table 3-2. Quality assurance objectives for In Vitro analysis.

Radionuclide	Minimum Detection[c] (Bq/Sample)	Precision[a]	Accuracy[b]
^{238}Pu	1×10^{-3}	0.40	-0.25 ±0.50
$^{239, 240}Pu$	1×10^{-3}	0.40	-0.25 ±0.50
^{234}U	1×10^{-3}	0.40	-0.25 ±0.50
^{238}U	1×10^{-3}	0.40	-0.25 ±0.50
^{90}Sr	1×10^{-2}	0.40	-0.25 ±0.50
^{137}Cs	1×10^{-2}	0.40	-0.25 ±0.50

a. The precision represents the relative dispersion of the values of accuracy from its mean for quality control samples (see definition) processed over one year.

b. The accuracy is defined as the agreement between result and the true value of quality control samples processes over one year.

c. MDAs are calculated by formulas given in Section 12.2.4.

4. SAMPLING PROCEDURES

Sample collection procedures are not applicable to RA laboratory activities. Sample collection is the responsibility of organizations sending samples to RA laboratories.

5. SAMPLE CUSTODY

Samples that are collected for legal purposes (e.g., environmental regulatory) are maintained under chain-of-custody (COC) in the RA laboratories from time of receipt to final disposal. Each laboratory has a sample tracking system to track all samples through the laboratory from receipt to disposal.

The ALD manager or the GL assigns sample custodians at each of the RA laboratory facilities to coordinate sample management activities. These sample custodians receive samples, store and track samples within the laboratories, maintain COC when required by the customer, and dispose of samples in accordance with company procedures.

Samples received at ALD laboratories must be accompanied by sufficient information from the requestor to allow the laboratories to dispose of residual samples and analytical residues in compliance with Federal, State of Idaho, and company regulations and practices.

Each laboratory assigns a unique identification number to each sample upon receipt. Sample handling processes are described in the following management control procedures (MCPs) and analytical chemistry laboratory procedures (ACLPs):

INTEC laboratories (RC and GS): MCP-2002, "Analytical Sample Management"
CFA laboratories (IVA and GS): ACLP 5.100, "Sample Receipt"
ACLP 5.300, "Sample Tracking"
TRA laboratories (RC and GS): ACLP-10.10, TRA Radioanalytical Sample Management

Samples requiring COC are stored in limited-access custody areas until analysis and after analysis has been completed. RA samples, with the exception of In Vitro samples, are stored at room temperature. In Vitro fecal samples are stored in a freezer at <0° C. Urine samples may be refrigerated, but must not be stored in a freezer. Any special sample storage requirements specified in project-specific requirements documentation are followed.

The analytical holding time for environmental/regulatory samples is six months from sample collection, unless otherwise specified in project-specific requirements documents.

At a minimum, samples are maintained at the laboratories until analyses have been completed and data have been approved for release. At customer request, environmental/regulatory samples may be maintained at the laboratory until customer validation of the reported data has occurred. Customers must provide a written justification for the laboratories to hold any sample longer than one year from reporting.

6. CALIBRATION PROCEDURES AND FREQUENCY

Calibration (see definition) of radiation detection systems is essential for making quantitative determinations from the measurement of radioactivity. Most detection systems are only capable of

detecting a fraction of the radioactivity actually being emitted from radioactive nuclides. Therefore, it is imperative to establish the relationship between the measured counting rates and that of the known emission (disintegration) rates. Such a relationship is commonly referred to as the detector efficiency (efficiency = counts per minute/disintegrations per minute [cpm/dpm]). In order to determine detection efficiencies, it is necessary to use calibration standards/sources that are well characterized and traceable to a national reference laboratory.

6.1 General Requirements

This section identifies general calibration requirements for the analytical instrumentation and measurement and test equipment (M&TE) (see definition) used in the RA laboratories. It includes standard traceability (see definition), environmental controls, calibration procedures, out-of-tolerance conditions (see definition), calibration status, storage and handling, selecting M&TE measurement standards (see definition), and calibration intervals.

6.1.1 Standard Traceability

All measurement standards used for instrument and M&TE calibration must be traceable to one of the following sources, as appropriate: a national or international certifying agency, a nationally recognized fundamental or natural physical constant, primary or secondary standards (see definitions), ratio calibrations, certified reference materials (see definition), or consensus standards (see definition). All measurement standards must be evaluated for accuracy, stability, range, and resolution by personnel responsible for calibrations to ensure adequacy of the standards for their intended use.

6.1.2 Environmental Controls

Measurement and test equipment and measurement standards are calibrated and utilized in an environment controlled to the extent necessary to ensure continued measurements of required accuracy, giving due consideration to temperature, humidity, vibration, cleanliness, or other controllable factors.

6.1.3 Calibration Procedures

Calibration procedures must, as a minimum, specify the measurement standards or equipment used, the required parameter, range, the required accuracy of the measurement, and the acceptable tolerance (see definition) of each measurement parameter.

Calibration procedures for the liquid scintillation systems, low energy photon spectrometers, gas proportional instruments, alpha spectrometers, gamma spectrometers, and analytical balances are described in Sections 6.2 through 6.7 of this document. Methods used to calibrate each instrument are described in the *Analytical Chemistry Laboratory Procedures Manual*; the *Analytical Chemistry Methods Manual* (ACMM); and INEEL/INT-99-00715, *Gamma and Alpha Analysis User Guide for Sun Sparcstations*.

Procedures for calibrating M&TE are maintained by the INEEL Standards and Calibration Laboratory (S&CL).

6.1.4 Out-of-Tolerance Conditions

When M&TE or measurement standards are found to be out-of-tolerance (see definition), the technical leader and the instrument custodian must be notified. The M&TE or measurement standard is removed from service by posting a notice on the instrument until the problem is corrected and the instrument recalibrated. An evaluation of all out-of-tolerance conditions must be performed by analytical personnel to determine the validity and acceptability of previous test results. Any M&TE or measurement standard consistently found out-of-tolerance is repaired or replaced. A calibration is performed when the accuracy of any M&TE or measurement standard is suspect.

6.1.5 Calibration Status

All M&TE and measurement standards must be labeled to indicate the status of calibration according to MCP-2391, "Control of Measuring and Test Equipment." This label must be attached to the M&TE or measurement standards and include the date calibrated. M&TE and measurements standards calibrated with use must be labeled accordingly and do not require a calibration date or calibration due date. If there is no label, the instrument does not need calibration.

6.1.6 Storage and Handling

Measurement and test equipment and measurement standards must be handled and transported in a manner that will not adversely affect the calibration or condition of the equipment giving due consideration to temperature, humidity, vibration, cleanness, or other controllable factors.

6.1.7 Intervals of Calibration

Instrument calibration minimum frequency intervals for routinely determined analytes are shown in Table 6-1. Calibrations are performed more frequently if indicated by instrument check source (see definition) data or quality control sample (see definition) analyses. The "With Use" notation in the Calibration Frequency column of the table is for nonroutine analyte analysis. The nonroutine analyte calibration is valid for one year unless indicated otherwise by instrument check source data or quality control sample analysis.

Table 6-1. Nuclear counting instrument calibration frequency.

Analyte/Technique	Instrument Total Calibration	Minimum Frequency for Calibration Checks
Tritium Liquid Scintillation	Initially	Annually
^{14}C Liquid Scintillation	Initially	Annually
^{85}Kr Liquid Scintillation	Initially	With use[a]
$^{226}Ra/^{228}Ra$ Liquid Scintillation	Initially	With use[a]
^{59}Fe Liquid Scintillation	Initially	With use[a]
^{63}Ni Liquid Scintillation	Initially	With use[a]
^{241}Pu Liquid Scintillation	Initially	With use[a]
^{129}I Low Energy Photon Spectrometry	Initially	Annually
Alpha Gas Proportional	Initially	Annually
Beta Gas Proportional	Initially	Annually
^{90}Sr/Total Sr Gas Proportional	Initially	With use[a]
^{90}Y Gas Proportional	Initially	Annually
Actinides Alpha Spectrometry	Initially	Annually
Gamma Spectrometry	Initially	Annually

a. For nonroutine analyte analysis.

6.2 Liquid Scintillation Instruments

6.2.1 Instrument Descriptions

Beckman models 6500, 6000 and Packard models 1900, 2500 CA Liquid Scintillation Systems are used for the analyses of ^{3}H, krypton (Kr)-85, carbon (C)-14, ^{241}Pu, and various other beta-emitting radionuclides. Each system consists of specialized signal processing/coincidence background suppression circuitry and an automatic sample changer.

6.2.2 Schedule for Calibration

Total calibration (see definition) of an instrument is performed initially and whenever calibration checks, check source data, or quality control sample results indicate the system calibration is out of tolerance. Total calibrations for ^{3}H, ^{85}Kr, ^{14}C, and ^{241}Pu are described in Section 6.2.3. When necessary, total calibrations for other isotopes are performed each time the isotopes are determined or when the previous calibration is more than one year old.

Calibration checks are performed annually using a set of primary standards (see definition), or more frequently if indicated by check source data or quality control sample results. Calibration checks are described in Section 6.2.4.

A check source is counted daily with use or with each set of samples analyzed. The check source evaluations are described in Section 6.2.5.

6.2.3 Total Calibrations for ^{3}H, ^{85}Kr, ^{14}C, and ^{241}Pu Analyses

The liquid scintillation counter calibration establishes the relationship between the counting efficiency of the instrument and the quenching characteristics of the sample. A quench calibration is performed by measuring the counting efficiencies of a series of standard solutions that have a known quantity of a radionuclide and are chemically quenched to differing degrees. An efficiency curve is generated and a curve fit is performed, which results in an equation of efficiency as a function of quench.

A commercial set of traceable quenched tritium standards and/or ^{14}C standards are used for calibration for these isotopes. If commercial standards are not available, standards with varying degrees of quench can be prepared from a traceable standard. Examples of this are described in the ACMM-5011, "Tritium Determination in Urine," and ACMM-5945, "Determination of Pu-241 in Environmental/Bioassay Samples." Each calibration standard is counted long enough so that the uncertainty of the count is the same or less than the uncertainty associated with the certified value.

6.2.4 Calibration Checks

Calibration checks are performed to verify that the relationship between the counting efficiency of the instrument and the quenching characteristics of a standard have not changed.

Traceable standards of tritium and ^{14}C are used for calibration checks. If commercial standards are not available, they can be prepared from standard reference material or obtained from the ALD Quality Control Laboratory.

Each of the quenched standards are counted and analyzed as samples and the result compared with the known standard value. The measured and known values are compared, and if they agree within 10%, the existing calibrations are verified. If this test fails, an additional calibration check is performed. Potential problems are investigated, corrective actions are initiated, and a calibration check and/or a total calibration is performed as appropriate.

6.2.5 Check Source Evaluation

Each liquid scintillation instrument automatically performs a system verification assessment daily and/or at the end of the sample set being analyzed. Vendor-supplied primary standards of tritium and ^{14}C are maintained in each system's sample changer for this purpose. A message indicating successful or unsuccessful verification assessment is printed at the system printer. If a failure is reported, the system is taken out of service and the manufacturer is consulted to determine system operability.

6.2.6 Calibrations for Other Beta Emitters

Standards are prepared in a manner similar to that described in the ACMM-5011, "Tritium Determination in Urine." Each calibration standard is counted long enough so that the uncertainty of the count is the same or less than the uncertainty associated with the certified value. Instrument calibration efficiency as a function of the degree of quench is calculated from the ratio of the observed instrument response to the standard value.

6.3 Gas-Flow Proportional Instruments

6.3.1 Instrument Descriptions

Gamma Products G5000, Canberra Model 2400 Series, Tennelec LB 4000, and Tennelec LB 5100 gas-flow proportional instruments are used to measure the gross alpha, gross beta, total Sr, ^{90}Sr and yttrium (Y)-90 content of prepared samples or filters. These instruments are designed for the simultaneous analyses of beta- and alpha-emitting radionuclides deposited on a 2-inch diameter stainless steel planchet or filters.

6.3.2 Schedule for Calibration

The instruments are calibrated initially for the determinations of gross alpha, gross beta, ^{90}Sr and ^{90}Y. Total calibrations for other isotopes are performed whenever they are being determined. The total calibrations for the determinations of gross alpha, gross beta, ^{90}Sr, and ^{90}Y are described in Sections 6.3.3, 6.3.4, and 6.3.5, respectively.

Calibration checks are performed annually, or more frequently if indicated by check source data or quality control sample results. Calibration check standards are developed in the laboratory using traceable standards of ^{241}Am, ^{238}Pu and ^{137}Cs. The standards are analyzed as samples and the results compared with the known standard value. To verify the existing calibration, the calibration check data must agree with the initial calibration within its associated 3-sigma error. If the calibration check is not successful, potential problems are investigated, corrective actions are initiated, and a calibration check or a total calibration for gross alpha, gross beta, Sr, ^{90}Sr, and ^{90}Y is performed as appropriate.

Check sources of ^{137}Cs and ^{241}Am, ^{210}Po or ^{238}Pu are counted weekly or prior to a sample analysis to verify proper operation of the gas proportional instruments. These system checks are described in Section 6.3.6.

Quality control samples are analyzed with each batch of samples. A successful quality control sample analysis result verifies the instrument performance, as well as the laboratory method and the analyst's proficiency. Criteria for an acceptable quality control analysis result is described in Section 9 of this document. If the quality control analysis result is not successful, corrective action is taken, followed by a calibration check and/or total calibration if it is instrument related.

6.3.3 Calibrations for Gross Alpha Determinations

Total calibration of the gas proportional counters is performed to determine the counting efficiency for alpha-emitting radionuclides. Alpha particles are easily attenuated by solids. As a result, the calibration should establish the instrument response as a function of the amount of solids that typically exist in unknown samples.

The instruments are calibrated using sets of planchets, each set having the same amount of alpha standard, but with varying amounts of solids. The standards are prepared by pipetting a known amount of the ^{241}Am or ^{238}Pu standard together with a known amount of solids (sodium nitrate solution) on the planchet and increasing the volume to a total of 5 milliliters. The liquid on the planchet is then evaporated under a heat lamp until dry. The planchets are counted for 50 minutes or until a counting uncertainty of 2% is obtained. The system alpha efficiency, as a function of deposited mass, is determined from the ratio of the instrument response to the known amount of standard on the planchet. An equation of efficiency versus weight is then fit to the data.

Detailed descriptions of the calibration procedure are given in the appropriate ACMM methods or ACLPs, e.g., ACMM-5300 "Determination of Gross Alpha and Gross Beta."

6.3.4 Calibrations for Gross Beta Determinations

Total calibration of the gas proportional counters is performed to determine the counting efficiency for beta-emitting radionuclides. Beta particles are attenuated by solids. As a result, the calibration must establish the instrument response as a function of the amount of solids that typically exist in unknown samples.

The instruments are calibrated using sets of planchets, each set having the same amount of ^{137}Cs, but with varying amounts of solids. The standards are prepared by pipetting a known amount of the ^{137}Cs standard together with a known amount of solids (sodium nitrate solution) on the planchet and increasing the volume to a total of 5 milliliters. The liquid on the planchet is then evaporated under a heat lamp until dry. The planchets are counted for 50 minutes or until a counting uncertainty of 2% or less is obtained.

The system beta efficiency, as a function of weight of solids, is determined from the ratio of the instrument response to the known amount of standard on the planchet. A data plot of the inverse of efficiency versus weight and a straight line regression equation is determined. If a positive slope cannot be determined from the data points, less any outlier(s), and associated sigma errors of the individual data points overlap, an average efficiency value is used.

Detailed descriptions of the calibration procedure are given in the appropriate ACMM methods or ACLPs, e.g., ACMM-5300 "Determination of Gross Alpha and Gross Beta."

6.3.5 Calibrations for Total Sr, ^{90}Sr and ^{90}Y Determinations

Total calibration of the gas proportional counters is performed to determine the counting efficiency for ^{90}Sr or ^{90}Y. Some of the beta particles are absorbed by the solids associated with the ^{90}Sr or ^{90}Y separations chemistry. As a result, the calibration of the instruments must establish the relationship between beta particle absorption and the weight of the solids on the prepared sample filter.

Multiple standards with the same quantity of ^{90}Sr or ^{90}Y and varying amounts of precipitate are prepared from a ^{90}Sr standard in secular equilibrium with ^{90}Y. The ^{90}Sr and ^{90}Y are chemically separated and precipitated as $SrSO_4$ or $NH_4YC_2O_4{\cdot}H_2O$, and each precipitate is weighed and then counted on each instrument for 50 minutes, or until a sufficient number of counts obtains a value varying less than 5% at 2 sigma. From the known quantities of ^{90}Sr and $SrSO_4$ or ^{90}Y and $NH_4YC_2O_4{\cdot}H_2O$ on the planchet and the measured counts per second, the efficiency for counting ^{90}Sr or ^{90}Y is calculated as a function of weight of solids.

Detailed descriptions of the calibration procedure are given in the appropriate ACMM methods or ACLPs, e.g., ACMM-5382, "Determination of ^{90}Sr."

6.3.6 Check Source Data

An alpha and beta check source (typically ^{137}Cs and ^{241}Am or ^{210}Po) are counted weekly or prior to sample analysis to verify proper operation of the gas proportional instruments. The observed count rates are compared to mean count rates that have been previously determined. The previously determined count rates are the average of multiple observations taken when the system was determined to be in calibration. The check source has failed if the observed count rate, corrected for radioactive decay, differs from the average count rate by more than 3 sigma or by 10%. The reason for the inconsistency is investigated, and any deficiencies are corrected. If check source failure persists for undetermined reasons, a calibration check or total calibration is required.

6.3.7 Calibration Source and Background Checks for 2π Windowless Alpha/Beta Chamber

The RML 2π gas-flow proportional counter chamber system, which is used to measure alpha and beta standards/sources, is performance checked prior to use on each day it is used with a ^{239}Pu or ^{137}Cs source. These measurement checks monitor the stability of the instrument and provide the response to radioactive sources. The observed source check must agree with the previous measurements corrected for decay within 3 sigma. If this criteria is not met, adjustments must be performed. The ambient background response of the instrument is also monitored prior to each use to ensure that there is no contamination or significant change in the background associated with the instrument.

6.3.8 Calibration Source and Background Checks for the RML Four-Channel Alpha/Beta Counter

The RML four-channel gas flow proportional alpha/beta counting system, which is used to measure fission rate monitor wires, is performance checked prior to use on each day it is used with a set of ^{90}Sr sources (one for each channel). These measurement checks monitor the stability of the instruments and provide the response to radioactive sources. The observed source check must agree with the previous measurements corrected for decay. If this criteria is not met, adjustments must be performed. The four-channel system is not normally used for alpha measurements; therefore alpha source checks are not performed. The ambient background response of the instruments are also monitored prior to each use to ensure that there is no contamination or significant change in the background associated with the instruments. Source and background checks for the four-channel detection system are recorded in the logbook assigned to the system.

6.4 Alpha Spectrometry

6.4.1 Instrument Description

The alpha spectrometry systems are computerized systems for pulse height analysis of alpha-particle-emitting radioactive samples using multiple detectors. Components of the systems include alpha spectrometer(s), signal shaping and handling electronics, data acquisition computer(s), host computer(s), data transport/archive units, and report printers.

The system software (NetSpec or AlphaVision) supports the concurrent operation of multiple detectors. The basic features are background correction, automatic peak gain adjustment, photopeak location and identification, interactive data reduction, and data report generation.

Operation of the systems is described in appropriate ACMM methods, ACLPs, and INEEL/INT-99-00715, or ORTEC A 36-B32, "Installation, User Interface, and Reference Guide, Alpha Vision®-32," identified hereafter as the "AlphaVision manual".

6.4.2 Schedule for Calibration

Each detector is initially calibrated for both energy and efficiency. The energy and counting efficiency calibrations are verified annually or more frequently if indicated by the internal sample standards or quality control sample analysis. Energy calibrations and efficiency calibrations are described in Section 6.4.3 and 6.4.4, respectively.

As the samples are prepared in the laboratory, a known amount of the appropriate tracer is added to determine chemical yield. The internal standard is discussed further in Section 6.4.5.

6.4.3 Energy Calibrations

A detector energy calibration is determined (kilo-electron volts [KeV]/channel) by comparing the observed and known energy lines from an appropriate calibration standard. System energy calibration is described in INEEL/INT-99-00715, or the AlphaVision manual.

6.4.4 Efficiency Calibrations

A detector efficiency calibration is determined from the ratio of the detection system response with a known standard value, i.e., a certified reference standard. The detector efficiency is determined in the NetSpec software by comparing the detector measurement(s) of the standard with the known activity of the standard. System efficiency calibration is described in INEEL/INT-99-00715, or the AlphaVision manual. The efficiency calibration is checked annually by counting a certified reference standard as a sample and comparing the analysis value with its associated standard value. The calibration is verified if the check calibration (see definition) value agrees within 3 sigma of the certified standard value. If the calibration check is not successful, potential problems are investigated, corrective actions are initiated, and a calibration check or a total calibration is performed as appropriate.

An overall counting efficiency is determined for each sample counted with an internal tracer. The internal tracer of known activity is added to each sample prior to chemical separation for the actinide of interest. The actinide and internal tracer is gathered from the sample with a neodymium fluoride precipitation onto a millipore filter for counting. The overall efficiency is then determined directly from the sample being analyzed using the ratio of the internal tracer activity (counts) observed and the known amount of standard activity added. This efficiency is used for the quantitative sample analysis and includes both chemical yield and detector efficiency. Operation of the system using an internal tracer is described in INEEL/INT-99-00715, or the AlphaVision manual.

6.4.5 Performance Verification

Performance of alpha spectrometry system is verified with each sample counted. Samples prepared in the laboratory for counting contain internal tracers, as described in Section 6.4.4. Energy calibration is verified and the detection efficiency is determined from the internal standard during data reduction and report generation.

Quality control standards are analyzed with each batch of samples. The results verify the laboratory methods, instrument performance, and analyst's proficiency. Criteria for an acceptable quality control analysis result is described in Section 9 of this document. If the result is not successful, corrective action is taken followed by a calibration check or total calibration as appropriate (if instrument related).

6.5 Low Energy Photon Spectrometry

6.5.1 Instrument Description

The detectors for low energy photon spectrometry (LEPS) are data linked to the (NetSpec or VAX) gamma spectroscopy system. The system performs pulse height measurements of photons and x-rays between 5 and 800 KeV emitted from radioactive samples using one or more detectors. Components of the system include free-standing planar germanium detectors, signal shaping and handling electronics, data acquisition computer(s), host computer(s), data transport/archive units, and report printers.

The software supports the concurrent operation of multiple detectors. The basic features include data acquisition control functions, detector calibration, background correction, photopeak location, photopeak fitting functions, nuclide identification, operator interactive data reduction, and data report generation.

The acquisition and analysis system supports both the LEPS and the gamma spectrometry. The LEPS system is used routinely for the measurement of iodine (I)-129 and nickel (Ni)-59. System operation is described in ACMM-3606, ACMM-5100, ACMM-5200, and INEEL/INT-99-00715.

6.5.2 Schedule for Calibrations

Each LEPS detector is initially calibrated for both energy and counting efficiency using a certified reference material. Calibrations for various geometries and matrices, including water, charcoal, and palladium iodide (PdI_2), are performed as required. Calibrations for ^{59}Ni fractions of dissolved and separated materials are performed on an as-needed basis. Routine calibration methods for charcoal and PdI_2 are described in Section 6.5.3 and 6.5.4; however, calibrations for other geometries/matrices may also be performed.

For a given detector, energy calibration is determined (KeV/channel) by comparing the observed and known energy lines from a ^{129}I or ^{59}Ni calibration standard. Relative energy calibrations are made using the 29 and 33 KeV x-ray lines and the 39 KeV gamma-ray line. Sufficient data is collected to effectively characterize each line. System energy calibration is described in INEEL/INT-99-00715, and procedure ACLP-10.41, "RML Germanium and LEPS Detector Calibration."

Primary standards are not available in the geometry configurations for most samples submitted for analysis. Secondary standards are prepared from primary standards by adding an appropriate amount of primary standard to the secondary standard matrix (see definition) material and mixing until homogenous.

Calibration of a LEPS for a specific detector geometry entails placing a secondary ^{129}I or ^{59}Ni standard at a prescribed source-to-detector distance, measuring the instrument response until the appropriate counting statistics are obtained, and calculating the detector efficiency as a function of the observed activity versus the known activity for a given geometry.

Annually, each detector calibration is checked for one or more of the geometries with an appropriate primary or secondary standard, and, if indicated, a recalibration for all geometries is performed. Initial efficiency calibrations are verified if the calibration check agrees within its 3 sigma uncertainty.

A commercially-supplied ^{129}I or ^{59}Ni check source is counted daily or just prior to the analysis of a sample. The daily check source program is described in Section 6.5.5

A quality control sample is analyzed with each batch of samples involving radioiodine chemical separation. Criteria for an acceptable quality control analysis result is described in Section 9 of this document. If the quality control analysis result is not successful, corrective action is taken followed by a calibration check or total calibration as appropriate (if instrument related).

6.5.3 Calibration for ^{129}I on Charcoal

A set of three charcoal ^{129}I secondary standards are prepared by the Analytical Operations QC Group of the Analytical Laboratories Department. The secondary charcoal standards are contained in a 2-inch diameter, 150-milliliter plastic vial filled to a depth of 1.75 inches.

Each vial is positioned on the LEPS detector in a fixed geometry and counted until an uncertainty of 5% or less in the 39.6 KeV photopeak area is obtained. From the known quantity of ^{129}I and the integrated photopeaks, the detector efficiency as a function of energy is calculated.

If the corresponding photopeak areas from the three standards of the same type agree within their 2-sigma uncertainty, the data are accepted for generation of a calibration curve. If they do not agree, the outlier is recounted. If the areas still do not agree, another standard is prepared at the same concentration and counted. This process continues until the data from three standards agree.

6.5.4 Calibration for ^{129}I in Water (PdI_2 Precipitate)

A set of three secondary standards are prepared as follows: (a) a known quantity of a traceable ^{129}I standard is added to each of three flasks containing water and varying quantities of carrier iodide; (b) the nominal ^{129}I activity of each of the three standards is 350 Becquerel (Bq) per second; (c) palladium chloride is added to each flask to quantitatively precipitate the ^{129}I as palladium iodide. Preparation of the PdI_2 is described in ACMM-3806, "Determination of ^{129}I." Each precipitate is collected, dried, weighed, and mounted in a reproducible configuration on a card.

Each precipitate is positioned on the LEPS detector in a fixed geometry and counted until an uncertainty of 5% or less in the area of the photopeak of lowest intensity (39.6 KeV) is obtained. From the known quantity of ^{129}I and the integrated photopeaks, the detector efficiency as a function of energy is calculated.

If the corresponding photopeak areas from the three standards agree within their 2-sigma uncertainty, the data are accepted for generation of a calibration curve. If they do not agree, the outlier is recounted. If the areas still do not agree, another standard is prepared and counted. This process continues until the data from three standards agree.

6.5.5 Daily Check Source Data

A ^{129}I or ^{59}Ni check source is counted daily or just prior to the analysis of a sample. Acceptable performance of the instrument is verified when the observed 39.6 or 6.7 KeV gamma energy agrees with the known energy and the observed disintegration rate agrees within 3 sigma of the previously determined check source value. The previously determined disintegration rates are the average of multiple consecutive observations taken when the system was determined to be in calibration. If either of these tests fail, the reason for failure is investigated, and any deficiencies are corrected. If check source failure persists for undetermined reasons, a calibration check or total calibration is required.

6.6 Gamma Spectrometry

6.6.1 Instrument Description

The gamma spectroscopy systems are computerized systems for pulse height analysis of gamma-ray-emitting radioactive samples using multiple detectors. The system performs pulse height measurements of photons between 50 and 3,000 KeV from samples using one or more detectors. Components of the systems include germanium detectors, detector shields, signal shaping and handling electronics, data acquisition computer(s), host computer(s), data transport/archive units, and report printers.

The software used supports the concurrent operation of multiple detectors. The basic features include data acquisition control functions, detector calibration, background correction, photopeak location, photopeak fitting functions, nuclide identification, operator interactive data reduction, and data report generation.

System operation is described in INEEL/INT-99-00715; EG&G-2533, VAXGAP: A Code for the Routine Analysis of Gamma-Ray Pulse-Height Spectra on a VAX Computer (EG&G Idaho 1988); EG&G-2672, Operators Guide for VAXGAP, a Gamma-Ray Spectrum Analysis Package (EG&G Idaho 1992), or ORTEC A 66-B2, "Software User's Manual, Gamma Vision®-32," (identified hereafter as the "GammaVision manual"), as applicable; and applicable ACMM and ACLP Manual procedures.

6.6.2 Schedule of Calibrations

Each gamma detector is initially calibrated for energy, peak width, and counting efficiency using certified reference materials for primary and secondary standards.

For a given detector, energy calibrations (KeV/channel) are done by a least squares fit the observed and known energy lines from an appropriate calibration standard. System energy calibrations are described in appropriate ACMM methods, ACLPs, and INEEL/INT-99-00715, or the GammaVision manual.

Annually, each detector calibration is checked for one or more of the geometries with an appropriate primary or secondary standard and, if indicated, a recalibration for all geometries is done. The initial efficiency calibrations are verified if the calibration check activity agrees within 3 sigma of the known value.

A check source is counted daily or prior to counting a sample to verify both energy and efficiency calibration. The check source program is described in Section 6.6.5.

6.6.3 Energy Calibrations

Energy calibrations, which are essential for radionuclide identification and quantitation from the measurement of radioactivity photopeaks, are determined by measuring a radioactive source with accurately known gamma-ray energies (covering the range of calibrations and measurements routinely performed). Energy calibrations are used to establish the relationship between the instrument channels (measured photopeak positions) and known gamma-ray energies.

6.6.4 Efficiency Calibrations with Primary and Secondary Standards

Standard counting geometries are efficiency-calibrated using commercially available primary standards (see definition). Primary standards are typically radionuclide reference materials, sources and standards traceable to accredited/certified national reference laboratories such as the National Institute of Standards and Technology (NIST-USA), the Physikalisch-Technische Bundesanstalt (PTB-Germany), and the National Physical Laboratory (NPL-United Kingdom). Secondary standards (see definition) are prepared from primary standards. Detector calibration for a given counting geometry (see definition) entails placing the standard at a specific source-to-detector distance and collecting spectral data in each energy line until at least the appropriate counting statistics are obtained. The detector efficiencies are determined by comparing the observed response (count rate) with the known activity of the standard. The calibration process is described in INEEL/INT-99-00715 or the GammaVision manual, as applicable. Examples of calibrated detector geometries are identified in Table 6-2; however, detector geometries are dynamic and can vary with changing customer requirements. A complete list of current detector geometries is kept on file at each GS laboratory.

Table 6-2. Examples of gamma-ray calibration geometries.

Sample Container	Distances From Detector	Sample Matrix	Type of NIST Standard
Ampule (5 mL)	10 cm	5 mL water	Primary
Point Source	10 cm	Encapsulated in plastic	Primary
Vial (50 mL)[a]	5 cm	1/8 in. water[b]	Secondary
Vial (50 mL)[a]	5 cm	1/4 in. water[b]	Secondary
Vial (50 mL)[c]	0 cm	1/4 in. soil	Secondary
Vial (150 mL)[c]	0 cm	1 in. soil	Secondary
Vial (150 mL)[c]	0 cm	2 in. soil	Secondary
Vial (150 mL)[c]	0 cm	2 in. charcoal (75 mL)	Secondary
Vial (150 mL)[c]	0 cm, 5 cm	1.5 in. water[d]	Secondary
Vial (BIO)[c]	0 cm	5 mL water[e]	Secondary
Vial (BIO)[c]	0 cm	10 mL water[e]	Secondary
Vial (BIO)[c]	0 cm	15 mL water[e]	Secondary
Vial (BIO)[c]	0 cm	20 mL water[e]	Secondary
Vial (LS)[f]	0 cm	10 mL water	Secondary
		20 mL water	
Marinelli (1.0 L)	0 cm	1.0 L water	Primary

a. Vial dimensions: 1-1/4 in. diameter, 3-1/4 in. long.

b. Geometry used for pelletized particulate filters.

c. Vial dimensions: 1-13/16 in. diameter, 3-7/8 in. long.

d. Geometry used for vegetation and tissue samples.

e. Geometry used for fecal ash samples.

f. Standard liquid scintillation glass vial.

6.6.5 Daily Check Source Data

An europium (Eu)-152 (or other nuclide or combination of nuclides) standard is counted daily with use or just prior to the analysis of a sample using each detector. The observed energy and disintegration rate for each selected gamma-ray line (e.g., 122, 344, 964, and 1408 KeV for ^{152}Eu) are compared to the known energy and the disintegration rate to validate the energy and efficiency calibration. Efficiency is validated if results are within 3 sigma. The previously determined disintegration rates are the average of observations taken when the system was determined to be in calibration. If either of these tests fail, the reason for failure is investigated, and any deficiencies are corrected. If check source failure persists for undetermined reasons, a calibration check or total calibration is required.

6.6.6 Instrument/Ambient Background Checks

Instrument/ambient background measurements are generally performed weekly on each gamma-ray spectrometer. The background measurement check frequency may be monthly if the following three conditions are met: 1) separate counting facilities are maintained for environmental samples; 2) specific programming allows for the use of multiple backgrounds; and 3) a documented history of background measurement stability exists. The counting duration for background assessments is generally 12 to 70 hours. Background full-energy peaks and their associated counting rates are evaluated to determine the level of stability of the background radiation and to ensure that no low-level contamination of the detector system has occurred.

6.7 Analytical Balances

6.7.1 Instrument Description

Analytical weight measurements are determined using commercially available, precision analytical balances and top-loading balances. The balance capacity, weighing units, and precision are generally inscribed on the instrument by the manufacturer.

6.7.2 Schedule for Calibration

Analytical balances are calibrated annually by the INEEL S&CL. This organization maintains calibration procedures and records for these activities.

Even though the S&CL performs an annual calibration, individual laboratory checks are performed by a built-in calibration feature of the balance or by use of Class-S NIST traceable weight sets. These checks are documented in the balance calibration/use logbook.

6.8 Validation of Computer Calculational Programs

Validation of computer calculational programs is performed per ALD MCP- 2009, "Control of Analytical Software." Multi-user computer systems and programs, as well as applications of commercially available software (e.g., spreadsheets and macros), are validated per ALD MCP-2009.

7. ANALYTICAL PROCEDURES

Analytical procedures and requirements for the RA laboratories are summarized in this section.

7.1 Routine Sample Analysis

Routine analyses in the RA laboratories are performed in accordance with written, approved analytical methods. Approved RA methods are included in the following volumes of the *Analytical Chemistry Methods Manual (ACMM)*:

- Volume II: Radiochemistry Laboratory Methods (INTEC)
- Volume VI: In Vitro Analysis Laboratory Methods
- Volume IX: TRA Radioanalytical Laboratory Methods

Methods in the ACMM include procedures for preparation of samples, radiochemical separations and determinations, water chemistry determinations, special reagent preparations, standard preparations, alarm procedures, and miscellaneous analysis procedures. Controlled procedures describing laboratory operational functions that do not directly involve sample analysis (e.g., sample tracking) are included in the Analytical Chemistry Laboratory Procedures (ACLP) Manual, Sections 3, 5 and 10. Control, review, and distribution of ACMM methods and ACLPs are described in ALD MCP-2001, "Control of Analytical Methods and Procedures." MCP-2001 requires that all methods and procedures be reviewed and approved by method authors, technical leaders, the Group Lead, the ALD QAO, and the Facility Manager. Reviews by company environmental affairs, and safety and health personnel are also obtained, as appropriate.

All ACMM methods and ACLPs are formally reviewed at least every five years. ACMM methods and ACLPs used by IVA are formally reviewed every two years. Existing methods and procedures may be revised within the formal review cycle to incorporate such desirable changes as improved chemical yields of analytes, reduction of hazards, reduction of wastes, and editorial changes clarifying the procedure. All method changes, except editorial ones, must be experimentally verified and documented per Section 7.3, and the revised method must undergo the same level of review and approval as the original method. All changes, including editorial ones, are issued as new revisions to the method or procedure.

Counting room personnel use approved methods in the ACMM, ACLP manuals, and INEEL/NT-99-00715.

7.2 Modification of Methods for Special Samples

Approved routine analysis methods must occasionally be modified for unusual sample matrices or conditions encountered during analysis. Method modifications must be approved by the appropriate technical leader (or designated alternate) and the customer and recorded in a laboratory notebook before the instructions are used in the analysis of samples. ALD MCP-2001 and ALD MCP-2008, "Analytical Data Recording, Review, and Reporting," provide further detail for documenting method modifications.

7.3 Verification of New Laboratory Methods

New methods may be selected and adapted from published methods or developed in the laboratory. Regardless of the source, new methods must be verified in a RA laboratory before they are used for the analysis of samples. Complete records of the verification of new methods are kept in permanently bound laboratory notebooks by the analyst(s) assigned to perform the selection, adaptation, or development of the methods.

A new method is first evaluated by analyzing synthetic samples prepared from matrix materials similar to the expected samples and known quantities of traceable radiotracers representing both analytes and expected interfering radionuclides. The method is modified as necessary until a successful analysis of the synthetic sample has been achieved. After successful analysis of a synthetic sample has been achieved, a minimum of five quality control samples in a matrix similar to the expected samples must be successfully analyzed according to the method. Records of the successful quality control sample analyses are maintained in laboratory notebooks and in the quality control records maintained by the ALD. After approval by the technical leader and hazard evaluation, the method may be used in the analysis of nonroutine samples. For use with routine samples, the method must be incorporated into the ACMM per procedures given in ALD MCP-2001.

8. DATA REDUCTION, VALIDATION, AND REPORTING

8.1 Data Reduction

Analysts are responsible for recording all sample data on the appropriate sample analysis work sheets, analytical logbooks, laboratory notebooks, or in other suitable documents such as instrument printouts. Calculations for data reduction are provided in ACMM methods. Most data reduction calculations are done by computer and all calculational algorithms are validated per ALD MCP-2009, "Analytical Software Control." In cases where hand calculations are required, the hand calculations are performed in duplicate by two different persons (i.e., 100% verification).

8.2 Data Validation

The data validation process at the generation level is described in MCP-2008. The validation process consists of multiple levels of technical and quality assurance review. All analysts are responsible for performing a completeness and accuracy check on their own data before passing it on to the next level of review. The analyst then turns the data over to the appropriate TL or a designated alternate for independent technical review. The person performing the independent technical review must be a technical peer who was not involved in the sample analysis or results calculation, and has appropriate education and experience to evaluate the technical accuracy and adequacy of the data. This person is

responsible for ensuring that data to be reported are accurate and complete per customer requirements (i.e., data for all requested samples and analyses, including required QC samples, are present).

The ALD QA officer or designee periodically performs QA review of data reports and packages. The frequency of QA review depends on customer or project requirements, and may be as frequent as 100%, but should not be less frequent than 5%.

8.3 Data Reporting

The data reporting process is described in ALD MCP-2008. Report formats and content vary with customer requirements. At a minimum, the measured result and associated uncertainty are reported for each requested analysis. Per project-specific requirements, reports may contain associated QC sample results and raw data. Based upon the customer's requirements, analysis results may be issued on specialized report forms, completed analysis request forms, as formal letters, as attachments to analysis request forms, internal technical reports, or laboratory information management system (LIMS) reports. At a minimum, all results must be approved (written signature-release or LIMS electronic approval) by the technical leader or a designated alternate (independent technical reviewer) before being submitted to the customer. Additional reporting procedures for the IVA group are described in ACLP-5.500, "Documentation and Record Storage" in Section 5 of the ACLP Manual.

8.3.1 In Vitro-Specific Reporting Requirements

In Vitro data are reported with both the estimated random and total uncertainties. Both the estimated random and total uncertainties are reported at one standard deviation. Small negative and other results less than or equal to the total uncertainty are interpreted as including zero or as not detected. For results greater than 2 times, but less than 3 times the total uncertainty, detection is questionable. The decision level, that quantity of analyte at or above which a decision is made that the analyte is definitely present, is 3 times the total uncertainty. Results greater than 3 times the total uncertainty indicate detection, i.e., are true positives.

8.4 Physical and Chemical Databases

The physical and chemical databases used in computerized and hand calculations applied to measured data must be verified and validated prior to their inclusion and use in any data analysis system or method. These databases include, but are not limited to, atomic numbers, atomic weights, molecular weights, molecular formulas, isotopic abundances, half-lives, decay modes, emission probabilities, emission energies, cross sections, valences, decay chains, and attenuation coefficients.

The information in the databases and any changes to them (i.e., additions, removals, or alterations) must be reviewed and approved by the technical leader or designated technical specialist before use. The databases currently used by ALD come from the latest evaluations that have undergone national and international review and testing. Independent and internal data specialists must agree that the databases are correct. Database correctness is validated based on comparisons of the results from calculations with "known" values.

If errors or inconsistencies are identified or observed in current databases, the technical leader or technical specialist is notified, and corrective actions as described in Section 13 are initiated. Any

sub-databases created for special applications must undergo the same level of technical review and approval as the initial database.

9. INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Quality control (QC) samples are routinely analyzed with samples to evaluate, establish, and monitor analytical method, instrument performance, and analyst performance. Quality control samples used to evaluate routine analytical method performance include blank samples (see definition), known standards, replicate samples (see definition), and spiked samples.

The type and number of QC samples analyzed differ for specific sample matrices and analytes. The QC samples are associated with actual samples through analytical batches (see definition). An analytical batch consists of twenty or fewer samples of similar matrix that are either analyzed simultaneously or processed sequentially on a continuous basis within the same working period (see definition). A working period may extend over several days when sample preparation and analysis procedures require it. For the IVA Group, Table 9-1 defines the types and frequency of QC samples analyzed with each batch of samples for the types of matrices and analytes. Quality control frequencies and types for other RA work are project specific and are specified in applicable project-specific QC requirements documents (e.g., statements of work).

Table 9-1. Reagent blanks and quality controls required in the analysis of In Vitro samples.

Sample Matrix	Analyte	Blank Samples	Matrix Blanks	QC Samples Water Solution	QC Samples Matrix
Fecal	^{90}Sr	1/set	None	1/set	None
Fecal	Uranium	1/set	None	1/set	None
Fecal	Plutonium	1/set	None	1/set	None
Fecal	^{241}Am	1/set	None	1/set	None
Urine	^{3}H	1/set[a]	Urine	1/set[c]	1/set
Urine	^{90}Sr	1/set[a]	Urine[b]	1/set[c]	1/set[b]
Urine	Uranium	1/set[a]	Urine[b]	1/set[c]	1/set[b]
Urine	Plutonium	1/set[a]	Urine[b]	1/set[c]	1/set[b]
Urine	^{241}Am	1/set[a]	Urine[b]	1/set[c]	1/set[b]

a. If a matrix blank is run, the reagent blank may be omitted.

b. When available.

c. If a matrix control (see definition) is run, the water control may be omitted.

9.1 Blanks

Blank samples are analyzed to detect potential contamination introduced during the analytical process due to chemical reagent impurities, laboratory equipment contamination, or sample cross-contamination. Blanks may also be measured to establish instrument backgrounds that may be altered by the blank matrix. Reagent blank samples are prepared from portions of the same reagents and tracers used in sample analyses. Matrix blanks are prepared from matrices similar to those of actual samples. Blanks may be introduced at various stages (e.g., sample dissolution, radionuclide separation, and counting) in the analytical process to monitor contamination associated with each stage. All blanks are handled and treated in the same manner as the actual samples.

Depending on project requirements, the result for the analyte in the reagent blank may be subtracted from the analyte content of the corresponding samples or reported separately as a QC sample. Acceptance criteria for blank results are dependent upon project requirements and the nature of the blank (i.e., where in the analytical process it was introduced). If only natural or added tracer levels of analytes are detected in the blank samples, the chemical reagents and analysis are shown to be free from contamination and cross-contamination. If a blank sample result does not meet acceptance criteria, the technical leader investigates the problem and documents the results of the investigation. Depending on project requirements, samples associated with the unacceptable blank may require reanalysis, or results may be reported with a qualification (e.g., data qualifier flag).

9.2 Control Samples

The accuracy and precision of the sample analysis results are verified by the analysis of control samples. Control samples, also known as laboratory control samples (LCSs), contain a known amount of specific analyte(s) taken from primary or secondary standards traceable to a standard reference material.

Control samples in water matrices are prepared by ALD personnel. Control samples in other matrices are prepared by adding water matrix control (see definition) samples to uncontaminated matrices similar to those of actual samples. The control samples are analyzed in the same manner as actual samples. Control samples are normally introduced into the beginning of the analytical process (e.g., sample preparation/dissolution). However, as with blanks, control samples can be introduced at various points in the analytical process to monitor the accuracy of separate steps in the process, if desired.

Control sample acceptance criteria are normally project specific. Acceptance criteria are generally expressed in one of three ways: (1) the result agrees with the known value within 2 sigma of the uncertainty of the result; (2) the result falls within two standard deviations of a historical control database mean; and (3) the result falls within a specified percent recovery range of the known value. If a control sample is unacceptable, the technical leader investigates to determine a cause and documents the results of the investigation. Depending on project requirements, samples associated with the unacceptable control sample may require reanalysis, or results may be reported with a qualification (e.g., data qualifier flag).

9.3 Replicate Samples

Replicate samples are multiple aliquots of the same sample that are processed separately through the analytical procedures. Replicate samples are analyzed for a variety of reasons: (1) when sufficient

sample is available, replicate analyses may be performed to verify the precision of the analysis system and the proficiency of the analyst; (2) when sufficient sample is available and when the sample matrix is not readily homogeneous, replicates may be performed to derive an average composition; and (3) when the sample preparation process is sufficiently complex such that there is a high potential to have poor yields or contaminate the sample, replicates may be processed to ensure that a satisfactory result is obtained. A single replicate (referred to as a laboratory duplicate) is often required for environmental/regulatory analysis QC. Replicate precision acceptance criteria are normally expressed as relative standard deviation, percent relative standard deviation, or relative percent difference (for two replicates). Because of the diverse applications of replicates, the use and acceptability of replicates is described in more detail in controlled analytical methods and project-specific quality requirements documents.

9.4 Spiked Samples

For most routine analyses, samples are individually spiked with radionuclide tracers (non-target analyte nuclides) to monitor analyte recovery through the analytical process. Depending on project requirements, sample analyte data may be corrected for tracer chemical yield before reporting data to the customer.

For some environmental/regulatory analyses, matrix spikes (MSs) or matrix spike duplicates (MSDs) are performed on one sample per analytical batch. Matrix spike and MSDs are prepared by adding target radionuclides to samples and carrying them through the analytical process. Matrix spike and MSD results are determined as percent recovery, and are reported separately from sample data. Sample data are not corrected for MS or MSD recovery.

9.5 Control Charts

Statistical control charts are generated and maintained for detector efficiency and background checks performed on each detector (see Section 6). If results of detector efficiency checks fall outside the 99% confidence interval around the mean, the detector is not used until appropriate corrective actions are taken (see Section 13).

10. PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

10.1 Internal Audits and Assessments

10.1.1 Self Assessments

ALD management participates in self-assessments per MCP-8, "Performing Management Assessments and Management Reviews." This program uses self-evaluation and feedback to identify areas or functions where improvement is needed or where excellence has been achieved in work activities. Results of self-assessments are documented and archived in ALD files.

10.1.2 Analytical Laboratories Department Quality Assessments and Surveillances

The ALD QAO or designee performs assessments and surveillances of RA activities to evaluate compliance with this QAPjP. These assessments focus on QA program implementation, process effectiveness, and data product quality.

A yearly assessment schedule is prepared to identify which activities and tasks are to be covered and the timeframe within which the assessments are to be performed. The assessment schedule and scope are reviewed periodically and revised as necessary to ensure that coverage is complete. A minimum of one assessment of RA activities must be performed annually.

10.2 External Audits, Assessments, and Surveillances

RA activities are subject to audits, assessments, and surveillances by multiple organizations external to the ALD. These assessment activities may cover all or part of the RA laboratory activities scope. The scope, scheduling, and performance of these assessment activities is the responsibility of the organization requesting or performing the assessment. Examples of external audits, assessments, and surveillances conducted on ALD or RA activities include:

- Multidisciplinary assessments by the INEEL Performance Assurance organization
- Technical and quality assessments by the Idaho Completion Project (ICP) Sample and Analysis Management (SAM) organization
- INEEL Environmental, Safety, Health, and Quality Assurance (ESH&QA) assessments of RC and IVA support to the Radiological Control Programs.

In addition to these external audits and assessments, external evaluation of RA performance is provided by the performance evaluation programs discussed in Section 3.5.

11. PREVENTIVE MAINTENANCE

Analytical instrumentation undergoes routine preventive maintenance as recommended by the equipment manufacturer or as dictated by experience. Maintenance schedules are established and included in analytical methods for analytical instrumentation. Maintenance procedures are incorporated by reference to the manufacturer's manuals or other ALD procedures (e.g., ACLPs).

Instrument custodians are appointed for major instrumentation systems, and are responsible for ensuring that required maintenance is performed and for maintaining an inventory of critical spare parts. Radioanalytical laboratories have sufficient redundant instrument capability to minimize downtime for a given analytical procedure. Critical spare parts provide backup of selected hardware most likely to fail. Software backups are maintained in fireproof cabinets, and hardware spare parts are stored in lockable cabinets. Backup software maintained includes duplicate copies of all the systems' software and current data. Redundant systems and systems with multiple detectors provide inherent backup capabilities. Additional hardware components that are maintained for system backup include spectroscopy amplifier(s), detector bias supply(s), analog-to-digital converter(s), mixer router(s), alpha particle detector(s), and NIM bin(s) with associated power supply.

The gamma and alpha acquisition and analysis system is a networked system with multiple alpha detectors and multiple gamma detectors. The computer work stations can operate with or independently of each other. The system includes germanium detectors for gamma-ray analysis and signal handling/shaping hardware that consists of preamplifier, amplifier, bias supply and analog-to-digital converter (ADC); and alpha spectrometry hardware consisting of an alpha spectrometer unit that consists of a detector, preamplifier, amplifier and silicon surface barrier detector with output signals to a mixer router and an ADC. Data are acquired, controlled, stored, and analyzed with computerized, user-interactive laboratory computer systems. These systems generate reports and graphics as required.

Nuclear counting facilities are maintained at laboratory temperatures within 70 ± 5° F. Regulated instrument power is used for the operation of voltage-sensitive instrumentation.

Maintenance logs and service logs/records are maintained for each of the instrument systems. Instrument serviceability records provide the failure rate of the various instruments.

12. SPECIFIC ROUTINE PROCEDURES FOR QUALITY ASSURANCE

Methods and calculations used to evaluate various quality control acceptance criteria are provided in this section.

12.1 Central Tendency and Dispersion of Data

12.1.1 Arithmetic Mean

The arithmetic mean of a data set is used to determine average sample concentrations when sample replicates are used to determine the average composition of a sample and to establish the center line for control chart generation. The arithmetic mean is calculated as follows:

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

where

$\bar{x}$ = arithmetic mean

x_i = i^{th} data point

n = total number of data points.

12.1.2 Standard Deviation

The standard deviation of a data set indicates the scatter about the mean and is used to estimate precision. The standard deviation is calculated as follows:

$$S = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$$

where

S = standard deviation

$\bar{x}$ = arithmetic mean

x_i = i^{th} data point

n = total number of data points.

12.1.3 Relative Standard Deviation

The relative standard deviation (RSD) is the standard deviation expressed as a fraction of the arithmetic mean, and is calculated as follows:

$$RSD = \frac{S}{\bar{x}}$$

12.2 Assessing Data Quality

12.2.1 Accuracy and Bias

Accuracy or bias are quantified using several different calculations, including percent recovery (%R), recovery ratio, relative difference, or relative bias.

Percent Recovery, when used to evaluate results of known laboratory control samples and standards and chemical yields, is calculated as follows:

$$\%R = \frac{C_m}{C_t} \times 100$$

where

C_m = the measured concentration of the known standard or tracer

C_t = the true (known) concentration of the standard or the amount of tracer added.

The ***recovery ratio*** is sometimes used to evaluate results of control sample analyses instead of percent recovery (particularly for PE program results) and is calculated as follows:

$$Recovery\ ratio = \frac{C_m}{C_t}$$

When ***percent recovery*** is used to evaluate results of matrix spikes or matrix spike duplicates, it is calculated as follows:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

SSR = measured concentration of spiked sample

SR = measured concentration of unspiked sample

SA = concentration of spike added.

Another expression of accuracy, ***relative difference***, is calculated as follows:

$$Relative\ difference = \frac{C_t - C_m}{C_t}$$

Relative difference is sometimes expressed as a percentage (above equation times 100) or an absolute relative difference (absolute value of the difference used in numerator).

Accuracy expressed as ***relative bias***, is calculated as follows:

$$Relative\ bias = \frac{C_m}{C_t} - 1$$

12.2.2 Precision

Precision between two replicate (duplicate) samples is expressed as the relative percent difference (RPD) and is calculated as follows:

$$RPD = \frac{|C_1 - C_1| \times 100}{\frac{C_1 + C_2}{2}}$$

where

C_1 = the original (first) measured sample result

C_2 = the duplicate (second) measured sample result.

Precision between three or more replicates is expressed as the relative percent standard deviation and is calculated as follows:

$$\%RSD = RSD \times 100$$

For sample analyses performed per SAM requirements, duplicate precision is expressed as the normalized absolute difference, and calculated as:

$$Normalized\ absolute\ difference = \frac{|S - D|}{\sqrt{(TPU_S)^2 + (TPU_D)^2}}$$

where

S = sample result

D = duplicate result

TPU_S = 1s total propagated uncertainty of the sample

TPU_D = 1s total propagated uncertainty of the duplicate.

12.2.3 Completeness

Completeness of the reported data (expressed as a percentage) is calculated as follows:

$$\%C = \frac{M_v}{M_t} \times 100$$

where

M_v = number of measurements judged to be valid (i.e., non-matrix affected QC samples in control)

M_t = total number of measurements requested (based upon number of samples submitted).

12.2.4 Detection Limits

The RA laboratories have adopted a definition and method of describing detection limits similar to that of L.A. Currie (Currie 1968). Detection limits are also referred to as minimum detectable activity (MDA) or minimum detectable concentration (MDC). The RA laboratories define detection limits as the lower limit at which a given analytical method (e.g., gamma-ray spectrometry) may be relied upon to produce a detection with a certain measure of confidence. It is that level at which there is 95% confidence that an activity will be detected above the background level. The detection limit indicates the capabilities of the entire measurement process under a given set of conditions. The MDA may be calculated using the following equation:

$$MDA\ (pCi/g) = \frac{2.71 + (4.65)s_b}{t \times E \times W \times 2.22 \times Y}$$

where

s_b = standard deviation of the background

t = count time (minutes)

E = detector efficiency (counts per disintegration)

W = sample aliquot weight (grams)

Y = fractional chemical yield.

The detection limit is influenced by many factors, such as the randomness of the radioactive decay process, the variability and fluctuations of background radiation, the sample geometry and matrix, the radionuclide mixture and concentration in the sample, sample volume, detector efficiency, and sample counting times. When these variables do not exist or can be minimized (controlled, held constant), conditions are considered "ideal." Detection limits determined under ideal conditions are referred to as *a-priori* (before the fact) detection limits and are usually based either on background or blank sample measurements. When these variables change (or fluctuate) significantly, *a-priori* detection limits no longer apply, and the detection limits are referred to as *a-posteriori* (after the fact). *A-posteriori* detection limits are usually based on the spectral conditions associated with the measurement.

12.3 Acceptance Criteria and Control Limits for In Vitro Analysis

12.3.1 Control Sample Acceptance Criteria

In Vitro analysis control sample results are acceptable if the absolute relative bias of the control sample result is less than twice the uncertainty of the ratio of the measured to the known value, that is:

$$\left|\frac{C_m}{C_T} - 1\right| < 2 \times \frac{C_m}{C_t}\sqrt{V_m^2 + V_t^2}$$

where

C_m = measured concentration

C_t = true (i.e., known) concentration

V_m = relative uncertainty of the measured value

V_t = relative uncertainty of the known value.

12.3.2 Control Charting Limits

Control charting limits for IVA control samples are calculated around the arithmetic mean of the twelve previous months' data as follows:

$$Control\ limits = \pm 3\sqrt{\frac{(Y/Z)}{(n-1)}}$$

where

$$Y = \sum_{i=1}^{n}\left(\frac{C_{m_i}}{C_{t_i}}\right)^2$$

$$Z = \frac{\left(\sum_{i=1}^{n}\frac{C_{m_i}}{C_{t_i}}\right)^2}{n}$$

C_m = measured value of the i^{th} QC sample

C_t = true (i.e., known) concentration of the i^{th} QC sample

n = number of measurements.

13. CORRECTIVE ACTION

Conditions adverse to quality are promptly identified and corrected as soon as possible. The cause of any significant condition adverse to quality (i.e. one that affects compliance with the QA/QC requirements of this document) is determined and action is taken to preclude its recurrence. The identification, cause, and corrective actions for conditions that do not comply with these quality requirements are documented and reported to appropriate levels of laboratory and customer management as indicated throughout this section.

The ALD corrective action system is part of the company system as described in company MCP-538, "Control of Nonconforming Items," and MCP-598, "Corrective Action System." The goal of the corrective action system is to detect and correct deficiencies before they become serious enough to cause data loss or the release of erroneous data.

Deficiencies identified as the result of internal or external audits, assessments or surveillances, performance evaluation programs, customer evaluations, external data validation, recurring analytical problems, control chart trends, or laboratory QA/QC are also subject to deficiency reporting and corrective action per MCP-598.

The technical leaders are responsible for immediately informing laboratory customers of any corrective actions that may impact the quality of previously reported data or current data to be reported.

14. QUALITY ASSURANCE REPORTS TO MANAGEMENT

The purpose of quality assurance reports to management is to apprise laboratory and customer management of RA support and QA activities. Quality assurance reports are prepared by the ALD QAO or an appropriate technical leader at least annually, and include, at a minimum, the following information:

- Identification and status summary of all initiated and on-going deficiency reports and corrective actions
- Identification of any significant or recurring QA/QC problems, recommended solutions, and corrective actions
- Results of any audits, assessments, or surveillances conducted during the period
- Summary of participation in and results of external performance evaluation programs
- Summary of revisions made to ALD controlled documents (ACMM Methods, ACLPs) related to RA activities
- Identification of needed revisions to this QAPjP
- Changes in personnel work assignments.

At a minimum, the quality reports to management are distributed to the ALD manager, the RA Group Leads, the ALD QAO, and all technical leaders. Management of major customer projects (e.g., SAM) may also be included in the distribution.

15. RECORDS MANAGEMENT

This section describes the storage and retention of records generated by or in support of RA activities. Radioanalytical records are handled in accordance with MCP-557, "Managing Records." PLN-1216, "Records Management Plan for the Analytical Laboratories Department," describes specific records management practices for the ALD. ACLP 5.500, "Documentation and Record Storage," supplements this document for the IVA Group.

Table 15-1 summarizes categories and types of QA records generated by RA activities. The minimum retention period for nonpermanent quality records is three years after the record becomes inactive. The minimum retention period for lifetime quality records is the operational life of the facility or item. All records generated by the RA laboratories are assigned disposition schedules per DOE Records Schedules. Retention times for ALD records are set to meet laboratory documentation and records requirements. The laboratory records system is not intended to fulfill regulatory records retention requirements for specific projects supported by ALD analytical capabilities; such records retention is the responsibility of individual project managers.

Records may be stored on paper, microfiche, or electronic media. Backup copies of computerized data are generated at least once a month, and the backups are stored in fire-resistant safes. QA records are stored in lockable filing cabinets or safes. Backup copies of calibration data are stored separately from the nuclear counting facilities.

Calibration records for M&TE calibrated by the INEEL Standards and Calibration Laboratory (S&CL) are maintained by S&CL.

A data file, designated by sample log number or data report number, is maintained for each set of samples. This file contains raw data associated with sample analysis, including copies of all nuclear counting instrument computer outputs, analysis work sheets, printouts of calculations of results and associated uncertainties, and the completed Analysis Request forms or other requested reporting forms. These records also identify the analyst(s) and the specific nuclear counting instruments and methods employed in the analyses. A summary of associated QC samples and their results is also kept in the sample data file.

Table 15-1. Categories and types of QA records generated by RA activities.

Category	Record Types	Classification
Audits, Assessments, and Surveillances	Audit, assessment, and surveillance plans, reports, responses, corrective actions, and final closures	Nonpermanent[a]
Calibration Records	Instrument calibration records, M&TE calibration records, calibration verification records, source check records, standard preparation records, standard certifications	Nonpermanent[a]
Computer Software Verification Records	Software documentation, software verifications	Lifetime[b]
Corrective Actions	Corrective action reports, deficiency reports, nonconformance reports, QA reports to management	Nonpermanent[a]
Data Reports	Data reports, reporting forms, letter reports, internal technical reports, data package reports	Nonpermanent[a]
Method Performance Data	Method development data, method performance verification data, detection limit data, control charts	Nonpermanent[a]
Performance Evaluation Programs	PE program data reports and raw data	Nonpermanent[a]
	PE program scoring reports	Nonrecord
Personnel Training and Qualification Records	Staff QA training records, method and technical training records	Lifetime[b]
Procedures	Quality plans, analytical methods, ACLPs, MCPs	Lifetime[b]
Raw Analytical Data	Analytical logbooks, notebooks, service & maintenance records, instrument printouts, sample preparation records, data reduction and calculation records, quality control data (measured and known values plus results), data review records	Nonpermanent[a]
Sample Tracking Records	Chain-of-custody documentation, analytical request forms, sample shipping and disposal records	Nonpermanent[a]

a. Minimum retention time for nonpermanent QA records is 3 years.

b. Minimum retention time for lifetime QA records is for operational life of the facility or item.

16. PERSONNEL TRAINING AND QUALIFICATION

Management ensures that personnel are adequately qualified and trained for their assigned functions including necessary education, training, technical knowledge, and experience. Management maintains and keeps available in the laboratory the relevant records of qualifications, training, skills, and experience of the technical personnel.

Training and qualification procedures used in the ALD are defined in ALD MCP-2006, "Analytical Laboratories Department Training and Qualification Program." Supplemental training procedures for IVA personnel are identified in ACLP-5.700. Before independently using an analytical method to analyze any routine samples other than quality control samples, ALD personnel must be trained and qualified to perform the analysis, and the training must be documented. To attain qualification on an analytical method, each analyst must receive appropriate training from the technical leader or another qualified analyst, including instruction on equipment operation, sample handling, method-specific concepts, safety considerations, data reduction and interpretation. Trainees must independently demonstrate proficiency by performing successful analysis of quality control samples. For nonroutine sample analysis, technical leaders may qualify an analyst based on demonstrated performance with nonroutine samples. The training basis must be documented, and training records are maintained per Section 15. An example of a training record for counting system operation is provided in Figure 16-1.

Quality control samples required for proficiency demonstrations for IVA group personnel qualification are specified in Table 16-1. In Vitro Analysis group personnel qualification is valid for 12 months after the successful completion of proficiency demonstration of QC samples. The period of qualification is extended for 12 months each year as long as the analyst has satisfactorily completed at least two quality control samples during the previous qualification period. An example of an IVA training record is shown in Figure 16-2. Qualification of an IVA analyst may be revoked if the percentage of acceptable results on quality control samples drops below 90%. The analyst may be reinstated by successfully completing the steps for the initial qualification certification.

Table 16-1. Qualification requirements for IVA methods.

Technical Procedure	Blanks[a]	Controls[b]
ACMM-5956: Preparation of Fecal Samples for Determination of Strontium and the Actinides	2[d]	10[d]
ACMM-5967: Preparation of Urine Samples for Determination of Strontium and the Actinides	2[d]	10[d]
ACMM-5978: Determination of Americium, Plutonium, and Uranium	2[d]	10[d]
ACMM-5945: Determination of ^{241}Pu in Environmental/Bioassay Samples	1	5
ACMM-5382: Determination of ^{90}Sr	2[d]	10[d]
ACMM-5903: Determination of Thorium	2[d]	10[d]
ACMM-5011: Tritium Determination in Urine	0	0
ACMM-5924: Determination of Total Uranium in Urine	2[d]	0[c]

a. The number of blanks that must be successfully analyzed to be certified for the specified analytical method.

b. The number of quality control samples that must be successfully analyzed to be certified for the specified analytical method.

c. No controls are available. To be certified, a trainee must witness a certified analyst perform the technical procedure twice and then perform the technical procedure under the supervision of a certified analyst on two separate occasions. Leaders/supervision may certify an analyst based on demonstrated performance with nonroutine samples.

d. Analyses must be satisfactorily completed in the form of at least two batches of six samples that are processed on separate days.

Employee Name: _____ Date: _____

This employee is qualified per MCP-2006 and PLN-153, Section 16 to perform the measurement of routine samples on the counting systems indicated below. This employee has received training on data reduction, interpretation and report generation for routine samples and routine data. This person is qualified to conduct quality control measurements and has demonstrated satisfactory proficiency in maintaining these measurement systems and recognizing normal and abnormal operating conditions.

	Date Qualified	Approval
I. Sample Control		
Is aware of sample logging, tracking and disposition requirements	_____	_____
II. Sample Measurement, Preparation, and Handling		
Knows radiological control requirements	_____	_____
Knows sample information input requirements	_____	_____
Knows sample mounting and counting requirements	_____	_____
III. Systems Operations		
Knows how to use computer systems	_____	_____
Knows how to handle and use liquid nitrogen	_____	_____
Knows how to operate/calibrate gamma spectrometer systems	_____	_____
Knows how to operate/calibrate alpha/beta proportional counter systems	_____	_____
Knows how to operate/calibrate liquid scintillation systems	_____	_____
Knows how to operate/calibrate alpha spectrometer systems	_____	_____
Knows how to operate/calibrate ion chamber systems	_____	_____
IV. Data Analysis and Reporting		
Knows how to analyze gamma-ray spectra	_____	_____
Knows how to analyze alpha spectra	_____	_____
Knows how to analyze gross counting data	_____	_____
Knows how to analyze liquid scintillation data	_____	_____
Knows how to report data	_____	_____

Approval Signatures:

Person Qualified: _____ Date: _____

Technical Leader: _____ Date: _____

Group Lead: _____ Date: _____

Figure 16-1. Example of counting systems operational training record

IVA METHOD TRAINING

_____ is qualified to perform the analysis of In Vitro samples according to the analytical methods dated below. The analyst has received appropriate laboratory instruction and, for those methods to which quality controls are applicable, satisfactorily completed the qualification requirements described in ALD MCP-2006 and PLN-153, Section 16 within the past twelve months.

This qualification is valid from _____to _____

Qualification Date	Analytical Method
	ACMM-5956: Preparation of fecal samples for Sr & actinides (Revision _____)
_____	Plutonium
_____	Uranium
_____	Americium
	ACMM-5967: Preparation of urine samples for Sr & actinides (Revision _____)
_____	Plutonium
_____	Uranium
_____	Americium
	ACMM-5978: Determination of Americium, Plutonium & Uranium (Revision_____)
_____	Plutonium
_____	Uranium
_____	Americium
_____	ACMM-5382: Determination of Strontium-90 (Revision_____)
_____	ACMM-5903: Determination of Thorium (Revision_____)
_____	ACMM-5011: Determination of Tritium in urine (Revision _____)
_____	ACMM-5924: Determination of Total Uranium in Urine Samples (Revision_____)

Approvals:

Employee Qualified_____ Date_____

Technical Leader_____ Date_____

Group Lead, Radioanalytical_____ Date_____

Figure 16-2. Example of IVA group analytical method training

17. PROCUREMENT

The procurement of goods and services is controlled to assure conformance with specified requirements. The Radioanalytical Groups implement INEEL (site-wide) contractor procedures in procuring goods and services. These procedures include: MCP-1185, "Acquisition of Materials," the guiding document for procurement of goods; and MCP-1186, "Service Acquisitions," for the procurement of services. Additionally, MCP-593, "Using Purchase Cards To Acquire Materials And Services," is the guiding document for using the "P-Card" system where off-the-shelf goods and services are deemed adequate.

18. REFERENCES

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MCP-561, "Quality Program Plan/Quality Assurance Project Plan Development."

MCP-557, "Managing Records.MCP-593, "Using Purchase Cards To Acquire Materials And Services."

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MCP-1185, "Acquisition of Materials."

MCP-1186, "Service Acquisitions."

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ORTEC, A 66-B2, "Software User's Manual, Gamma Vision®-32"

APPENDIX A

Definitions

Acceptable Tolerance—The permissible deviation of an instrument response from the true value being measured.

Analytical Batch—A batch of samples consisting of twenty or fewer samples and associated QC of similar matrix which are either analyzed simultaneously or processed sequentially on a continuous basis within the same working period by the same analyst.

Blank Sample—An analyte-free matrix that undergoes preparation and analysis processes identical to those used on actual samples. Reagent blank samples include only the reagents used in the procedure, while matrix blank samples include matrix material as similar to actual samples as possible. The blank sample is used to document the absence of contamination resulting from the laboratory sample preparation and analytical process or cross-contamination between samples. Matrix blank samples are analyzed whenever uncontaminated sample matrix material is available.

Calibration—The comparison of measurement and testing equipment (M&TE) or measurement standard of unknown accuracy to a measurement standard of known accuracy to detect, correlate, report or eliminate by adjustment any variations in the accuracy of the instrument or measurement standard being compared.

Check Calibration—The verification of the energy and efficiency calibration of an instrument for a specific radionuclide, counting geometry, matrix, and source-to-detector distance. The purpose of a check calibration is to ensure the instrument is unchanged since the last total calibration.

Check Source—A permanently encapsulated or fixed source of a known quantity of a specific radionuclide(s), not necessarily traceable to the National Institute of Standards and Technology (NIST). A check source is counted daily prior to the analysis of any sample to assure that the instrument response is unchanged since the last calibration.

Consensus Standard—An artifact or process that is used as a *defacto* standard when no recognized certified reference material standard is available.

Counting Geometry—The physical configuration in which a sample, standard material (see definition) or check source is placed on the detector of an instrument. For a given geometry, the distance between the sample, standard material, or check source and the detector is fixed by a holder designed to position the sample, standard material or check source container in a given reproducible orientation.

Decision Level—The amount of a count or final instrument measurement of a quantity of analyte at or above which a decision is made that the analyte is definitely present.

Instrument Method—An instrument-based analysis procedure that describes the calibration and operation of the instrument system rather than the determination of a specific sample constituent.

APPENDIX B

Matrix—Either the final composition and form of a standard material used for nuclear instrument calibration or the composition and form of a sample subjected to analysis. Example matrices include water, soil, air filters, precipitates, and electrodeposited materials.

Matrix Control—A QC sample of a known traceable analyte(s) content prepared in a matrix similar to the actual samples in order to simulate actual samples. Matrix quality control samples are analyzed concurrently with actual samples to verify the efficiency of the procedures and the proficiency of the analysts.

Measurement Standard—Those devices used to calibrate Measuring and Testing Equipment or other measurement standards and prove traceability.

Measuring and Testing Equipment (M&TE)—All devices used to measure, gauge, test, inspect, or otherwise determine compliance with prescribed technical requirements.

Out of Tolerance—The inability of M&TE or measurement standard to measure a known value within acceptable tolerances.

Quality Control Sample—A sample or standard analyzed to determine or verify the performance of methods, instruments, and analysts.

Replicate Samples—Separate aliquots from the same sample that are prepared and analyzed to verify the reproducibility of the procedures.

Standard Materials

> **Certified Reference Material**—A reference or standard, distributed by a recognized national or international certifying agency such as NIST, The New Brunswick Laboratory, or the International Atomic Energy Agency.
>
> **Primary Standard**—A permanently encapsulated source, or fixed source, or other material prepared by a documented procedure from a certified reference material. A primary standard may also be prepared from other material if the measured value is confirmed by two independent methods.
>
> **Secondary Standard**—A permanently encapsulated source, or fixed source, or other material prepared by a documented procedure from a primary source. A secondary standard may also be prepared by comparing its sample content to either a primary standard or certified reference material using a documented instrument method (see definition). Secondary standards are used for calibrations of nuclear counting instruments only if a suitable primary standard is unavailable.

Total Calibration—The determination of the efficiencies of an instrument over the full range of variables encountered in a given sample analysis. Such variables include radionuclide energy, sample weight, sample volume, sample matrix, and sample-to-detector distance.

APPENDIX B

Traceability—The ability to trace the history, application, or location or an entity by means of recorded identification. For measurement standards, this is the ability to relate individual measurement results through an unbroken chain of calibrations to one or more of the following:

- U.S. national standards maintained by NIST, the U.S. Naval Observatory, the DOE Albuquerque Operations Primary Standards Laboratory, the New Brunswick Laboratory, Amersham-England or the International Atomic Energy Agency
- Fundamental or natural physical constants with values assigned or accepted by NIST
- Primary or secondary standards
- National standards of other countries that are correlated with U.S. national standards
- Ratio type of calibrations
- Comparison to consensus standards.

In a data collection sense, traceability relates calculations and data generated throughout a project or analysis back to the requirements for quality by being able to follow a standardized chain of events with standardized documentation.

Working Period—A period of time spent performing analyses on a batch of samples. This should not exceed one month per batch.