1. ABSTRACT

This method describes determination of gamma-ray radionuclide activities in a variety of samples or sources. The samples are counted in standardized and calibrated geometrics on semiconductor-based gamma-ray spectrometry systems. The gamma-ray analyses of spectra from the measurements, with all pertinent sample information, are accomplished and stored on Radiation Measurements Laboratory (RML) computer systems. The computer analysis program locates photopeaks, directs photopeak fitting to specific gamma-ray energies, performs counting loss corrections, identifies possible radionuclides, makes error estimates, computes radioactivity, computes concentration of radionuclides, provides diagnostics, prints results, and/or prints reports.

2. APPLICABILITY

This method is used by the RML to determine the gamma-ray radionuclide activities in soil, vegetation, biotic, liquid, and point source samples received from various INEEL programs or customers. This method is based on the following standard ASTM methods, procedures and terminology.

A. E170, Terminology Relating to Radiation Measurements and Dosimetry.
B. E181, General Methods for Detector Calibration and Analysis of Radionuclides.
C. C1009, Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories within the Nuclear Industry.
D. C1068, Guide for Qualification of Measurement Methods by a Laboratory within the Nuclear Industry.

Appropriate sample containers approved for use with this method are:

A. Soil, vegetation and biotic samples – 500 cm3, 16 oz. plastic squat jar or a 70 cm3, 100 ml plastic pill vial.
B. Liquid samples – 540 or 60 ml plastic bottles, and 1 or 4 liter marinelli beakers.
C. Point Source – A source for which the dimensions are small and container density is low, allowing the detection efficiency in the measurement setup to simulate a point source.

A Job Safety Analysis (JSA) was developed for this procedure in accordance with a determination made using MCP-3562, *Hazard Identification, Analysis & Control of Operational Activities* and MCP-3480, *Environmental Instructions for Facilities, Processes, Materials and Equipment.*

3. DISCUSSION

The samples can be counted on any germanium (Ge) detector system in the RML having an efficiency calibration for the specific sample type and container.

In general, the efficiency calibration is performed per ACLP-10.41, *RML Germanium and LEPS Detector Calibration*, which consists of acquiring or preparing sources with certified radionuclide activities, in specific geometrics and counting them at known source-to-detector distances. The spectra are analyzed with the VAXGAP or PCGAP spectral analysis codes, to determine the full energy peak counting rate per emission rate over the energy range of application.

A detection efficiency versus energy relationship is established and used to deduce the activities of the sample being analyzed.

Analyses performed are based on customer-defined requirements that are usually contained in statements of work, task order statements, or task authorization agreements.

4. SAFETY PRECAUTIONS

4.1 Radioactive Materials And Sample Hazards

4.1.1 Handle radiological samples as specified on the applicable Radiological Work Permit (RWP). (See MCP-7, *Radiological Work Permit*). Radiological Control Technician (RCT) coverage may be required when handling radiological samples. [JSA]

4.1.2 Use care to maintain personal radiation exposure as low as reasonably achievable (ALARA).

WARNING

The samples being analyzed may be highly radioactive [JSA]

4.1.3 Handle samples not previously identified as radioactive, that are determined to be radioactive, under the appropriate RWP. [JSA]

NOTE: *Samples in glass containers are not allowed in the RML without prior approval by RML supervision. [JSA]*

4.1.4 Store samples being analyzed in an appropriate radiological area when not being handled or counted to limit personnel exposure. [JSA]

4.2 Chemical Handling

Ensure sample(s) are properly sealed to avoid the spread of contamination. [JSA]

4.3 Instrument Hazards

Keep hands clear of moving parts and pinch points and operate the moveable shielding with caution, when placing sample(s) on a turntable or in a holding device. [JSA]

5. APPARATUS AND REAGENTS

5.1 Apparatus

5.1.1 Standardized sample containers.

5.1.2 Detector system consisting of a high resolution Germanium (Ge) spectrometer with a cryostat, which maintains the detector crystal in a vacuum at cryogenic temperatures, a liquid nitrogen dewar, and associated electronics including a low noise preamplifier and high-voltage filter.

5.1.3 NIST traceable Eu-152 point source.

5.2 Reagents

None

6. SAMPLE HANDLING

Store biotic samples in an approved freezer prior to analysis. Return them immediately to an approved freezer after analysis, and do not open without IH review. [JSA]

7. PROCEDURES

NOTE: *All sections are not required to be performed. All steps within a section are to be performed in sequence unless other instructions are provided.*

7.1 Counting Procedure for Soil, Vegetation, and Biotic Sample

7.1.1 Laboratory Analyst: Ensure the required energy calibration measurements have been made and properly entered in the analysis routines per ACLP-10.41, *RML Germanium and LEPS Detector Calibration.*

7.1.2 Ensure the sample is properly sealed to reduce the possibility of contaminating personnel or equipment. [JSA]

7.1.3 Keep hands clear of moving parts and pinch points and the moveable shielding must be operated with caution, when placing sample(s) on a turntable or in a holding device.

7.1.4 IF appropriate, place the sample on a turntable (used to rotate the sample during counting)
THEN slide the turntable flush to the stop.

7.1.5 When placing sample(s) on a turntable or in a holding device, hands must be kept clear of moving parts and pinch points and the moveable shielding must be operated with caution. [JSA]

NOTE: *Count time is based on customer-required detection limits.*

7.1.6 Start the count.

7.1.7 WHEN the count is finished, save the gamma-ray spectrum.

7.1.8 Run the appropriate spectral analysis routines, as required.

7.2 Counting Procedure for Liquid Samples

7.2.1 Laboratory Analyst: Ensure the required energy calibration measurements have been made and properly entered in the analysis routines per ACLP-10.41, *RML Germanium and LEPS Detector Calibration.*

7.2.2 Ensure the sample is properly sealed to reduce the possibility of contaminating personnel or equipment. [JSA]

7.2.3 Keep hands clear of moving parts and pinch points and the moveable shielding must be operated with caution, when placing sample(s) on a turntable or in a holding device.

7.2.4 Place the samples in the appropriate sample holder for detector being used.

7.2.5 Place 1- and 4-liter marinelli beakers over detector housing on uplooker detector systems. (Make sure the marinelli is resting on the support platform and not the detector housing.)

NOTE: *Count time is based on customer-required detection limits.*

7.2.6 Start the count.

7.2.7 WHEN the count is finished, save the gamma-ray spectrum.

7.2.8 Run the appropriate spectral analysis routines, as required.

7.3 Counting Procedure for Point Source Samples

7.3.1 Laboratory Analyst: Ensure the required energy calibration measurements have been made and properly entered in the analysis routines per ACLP-10.41, *RML Germanium and LEPS Detector Calibration.*

7.3.2 Ensure the sample is properly sealed to reduce the possibility of contaminating personnel or equipment. [JSA]

7.3.3 Determine and establish the required geometry and distance using established efficiency tables.

7.3.4 Keep hands clear of moving parts and pinch points and the moveable shielding must be operated with caution, when placing sample(s) on a turntable or in a holding device.

7.3.5 Position the sample appropriately for the detector being used and start the count.

NOTE: *Count time is based on customer-required detection limits.*

7.3.6 WHEN the count is finished, save the gamma-ray spectrum.

7.3.7 Run the appropriate spectral analysis routines, as required.

8. QUALITY CONTROL REQUIREMENTS

8.1 Energy Calibration of Spectrometer Systems

Perform energy calibrations per ACLP-10.41, *RML Germanium and LEPS Detector Calibration.*

8.2 QC Standard Check for Spectrometer Systems

8.2.1 Check the efficiency of each spectrometer each calendar month, or when necessary, to verify the detection efficiency reproducibility and the energy resolution at low, medium and high energies using a NIST traceable Eu-152 point source.

8.2.2 Count the standard for 30 minutes on all spectrometer systems.

8.2.3 IF results disagree from prior results 5% or more, THEN a reason for the difference must be evaluated.

8.3 Ambient Background Check of Spectrometry Systems.

Instrument background counts, typically of 12 hour counting duration are accumulated monthly on each spectrometer system. Background photopeaks and their associated counting rates are evaluated to determine the level and stability of the background radiation and to assure that no low-level contamination of the detector system has occurred. The background spectrum permits a background correction to be made on subsequent spectra. Background spectra are also measured for blank water to use with liquid sample analyses where the presence of water alters the backgrounds.

9. CALCULATIONS

The radioactivity calculations for this method are accomplished by the RML computer systems and a derivative of the Gamma Analysis Program (GAP) computer software.

10. RECORDS

Records Description	Uniform File Code	Disposition Authority	Retention Period
Data printouts Data reports Electronic data Electronic raw spectral data results file	7101	See MCP-2007, *Analytical Records Management*	

11. REFERENCES

11.1 ACLP-10.41, RML *Germanium and LEPS Detector Calibration*

11.2 An Operator's Guide for VAXGAP, *A Gamma-Ray Spectrum Analysis Package*, EGG-2672, August 1992.

11.3 ASTM Annual Book of Standards, Volume 12.0 (E170, E181, C1009, and C1068)

11.4 Form 410.03, *Radioanalytical Services Analysis Request Form*

11.5 Gamma and X-Ray Spectrometry with Semiconductor detectors, New York: North-Holland, 1988, K.Debertin, R.G. Helmer

11.6 MCP-7, *Radiological Work Permit*

11.7 MCP-2001, *Control of Analytical Methods and Procedures*

11.8 MCP-2007, *Analytical Records Manag*ement

11.9 MCP-3480, *Environmental Instructions for Facilities, Processes, Materials and Equipment*

11.10 MCP-3562, *Hazard Identification, Analysis & Control of Operational Activities*

11.11 MCP-3635, *Chemical Hygiene Plan*

11.12 NCRP Report No. 58, *A handbook of Radioactivity Measurements Procedures*

12. SUPPLEMENTAL INFORMATION

12.1 History of ACMM-3606:

Revision	Author(s)	Date
0	Ron Murray/Gene Taylor	May 2000
1	Ron Murray	February 2002

12.2 Revision Summary

Revision 1 includes miscellaneous editorial changes and Section 4, Safety Precautions. Steps were reworded to contain active action verbs, and Appendix A was restructured to individually address the particular source requirements.

13. APPROVAL SIGNATURE BLOCK

POSITION TITLE	SIGNATURE	DATE
Method Author		2-5-02
Responsible ALD Tech Leader		2-5-02
Responsible ALD Supervisor		2/6/02
ALD QA Officer		02/05/2002
ALD Manager		2/5/02
ALD Facility Manager		2-5-02

Analytical Method Analytical Laboratories Department	**GAMMA-RAY ANALYSIS OF STANDARDIZED SAMPLES**	Identifier: ACMM-3606 Revision: 1 Page: **A1 of A1**

APPENDIX A

Procedure Basis

Step(s)	Basis/Summary	Source
4.1.1	Handle radiological samples as specified on the applicable Radiological Work Permit (RWP). (See MCP-7, *Radiological Work Permit*). Radiological Control Technician (RCT) coverage may be required when handling radiological samples.	JSA # ACMM-3606
WARNING	**The samples being analyzed may be highly radioactive.**	JSA # ACMM-3606
4.1.3	Handle samples not previously identified as radioactive, that are determined to be radioactive, under the appropriate RWP.	JSA # ACMM-3606
NOTE	*Samples in glass containers are not allowed in the RML without prior approval by RML supervision.*	JSA # ACMM-3606
4.1.4	Store samples being analyzed in an appropriate radiological area when not being handled or counted to limit personnel exposure.	JSA # ACMM-3606
4.2	Ensure sample(s) are properly sealed to avoid the spread of contamination.	JSA # ACMM-3606
4.3	Keep hands clear of moving parts and pinch points and operate the moveable shielding with caution, when placing sample(s) on a turntable or in a holding device.	JSA # ACMM-3606
6	Store biotic samples in an approved freezer prior to analysis, return them immediately to an approved freezer after analysis, and do not open without IH review.	JSA # ACMM-3606
7.1.2	Ensure the sample is properly sealed to reduce the possibility of contaminating personnel or equipment.	JSA # ACMM-3606
7.1.5	When placing sample(s) on a turntable or in a holding device, hands must be kept clear of moving parts and pinch points and the moveable shielding must be operated with caution	JSA # ACMM-3606
7.2.2	Ensure the sample is properly sealed to reduce the possibility of contaminating personnel or equipment.	JSA # ACMM-3606
7.3.2	Ensure the sample is properly sealed to reduce the possibility of contaminating personnel or equipment.	JSA # ACMM-3606