RML GERMANIUM AND LEPS DETECTOR CALIBRATION	Identifier: ACLP-10.41 Revision: 1 Page: **1** of 12

Analytical Laboratories Department	Analytical Laboratories	For Additional Info: 208-533-4448	Effective Date: 04/02/2003
Manual: Analytical Chemistry Laboratory Procedures		**USE TYPE 2**	Change Number: 10.021

1. PURPOSE

This procedure provides instructions to energy calibrate, calibration check, and background check the Radiation Measurements Laboratory (RML) Ge Detector systems and low-energy photon spectrometer (LEPS) systems.

2. SCOPE

2.1 Energy Calibration of Spectrometer Systems

In order to accurately identify radionuclides, the gamma-ray spectrometer systems are calibrated with respect to photopeak energy. This calibration is performed with a source of well-known gamma-ray energies and a computerized calibration routine which establishes the photopeak energy versus spectrum channel and the photopeak width versus spectrum channel.

The gamma-ray spectrometer systems are energy calibrated for each detector system to establish the relationship between photopeak channel positions and actual photopeak energies (channel number versus gamma-ray energy).

The computer energy calibration method of pulser-equipped spectrometers determines pulser energy equivalents from a thorium source spectrum. These energy equivalents, along with continuously input pulser data during sample counting, provides for automatic and unattended energy calibrations with each spectrum.

The computer energy calibration method for non-pulser-equipped spectrometer systems finds the location of the 2614 keV gamma-ray and from its position calculates the energy per channel. With this energy per channel, the calibration program locates four other full-energy gamma-ray peaks, measures the peak position for all five photopeaks, and performs a least-squares quadratic fit of the resulting peak positions to their known energies in order to obtain the energy equation coefficients.

A least-squares fitting process is also performed using channel positions and the full-width-at-half-maximum of the peaks to determine the coefficients of the width equation. The peak position and width results from fitting the spectral data with a Gaussian function. A printed table is produced which shows the measured values and the values calculated with the fitting equation. The energy and width calibration coefficients are automatically stored with each analyzed sample spectrum. Each sample spectrum with its associated calibration information is stored on computer disk.

Spectrometer data slots can also be energy calibrated (i.e., the establishment of a relationship between the peak positions and corresponding energies) by using manually input values. This allows the operator to energy calibrate a spectrum by entering the location of a peak centroid and the corresponding energy.

2.2 Instrument or blank sample background counts, typically of 12-hour counting duration, are accumulated on each Ge or Ge(Li) gamma-ray spectrometer each calendar month and/or before and after each set of environmental samples. 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. It also allows noise and lower-level discriminator checks of the spectrometer electronics.
The background spectrum permits a background correction to be made on subsequent measurement spectra.

2.3 Equipment and Materials

2.3.1 For section 3.1:

A. RML computer system

B. Germanium detector system

C. NIST traceable thorium 228 source.

2.3.2 For section 3.2:

A. RML computer system

B. Low-energy photon spectrometer (LEPS) system

C. NIST source: Co-57, Sb-125, I-129, Ba-133, Eu-155, Am-241, or an appropriate x-ray emitting source.

2.3.3 For section 3.3:

A. RML computer system

B. Germanium detector system.

2.3.4 For section 3.4:

A. RML computer system

B. Germanium detector system

C. NIST europium 152 source.

2.4 A Job Safety Analysis (JSA) was developed for this procedure in accordance with a determination made using MCP-3562 and MCP-3480.

2.5 Safety Precautions

A. RCT coverage may be required when handling radiological sources as specified on the applicable Radiological Work Permit (RWP). [JSA]

B. Keep hands clear of moving parts and pinch points when introducing or removing source and holder to/from the detector, and operate moveable shielding with caution. [JSA]

3. PROCEDURES

NOTE 1: *Not all sections are required to be performed. All steps within a given section are to be performed in sequence unless other instructions are provided.*

NOTE 2: *The controls for sealed radioactive sources as defined in MCP-137, Radioactive Source Accountability and Control, are applicable during theperformance of this procedure.*

3.1 Ge Detector Energy Calibration

NOTE: *Energy Calibration is not required on detectors that are out-of-service (OOS).*

3.1.1 Lab Analyst: Perform a Ge detector energy calibration per this section based on the following criteria:

- Non-pulser-equipped detectors are energy calibrated prior to each shift's series of counts.
- Pulser-equipped detectors are energy calibrated once per week except for the sample changers (Ortec-15, Ortec-16, and RJG-3).
- The Ortec-15, Ortec-16, and RJG-3 sample changers are energy calibrated prior to use.
- An energy calibration may be performed anytime deemed necessary by the lab analyst.

WARNING

When handling sources, failure to minimize handling time, use shielding, or use remote handling devices such as handles or tongs may result in increased personnel exposure. [JSA]

3.1.2 When handling sources throughout this section, minimize handling time, use shielding, and use remote handling devices such as handles or tongs, as appropriate. [JSA]

3.1.3 Open the detector shield and place an energy calibration source (Th-228) in the source card sample holder.

3.1.4 Adjust the source holder to the proper source-to-detector distance.

NOTE: *The B7 (RJG-3) detector system has a source-to-detector distance of 0 cm. RML senior staff may recommend other source-to-detector distances (for other detector systems) for thorium energy calibration at their discretion.*

3.1.4.1 If using the B7 (RJG-3) detector system, adjust source-to-detector distance to 0 cm.

3.1.4.2 If using a detector system other than B7 (RJG-3), adjust source-to-detector distance to 10 cm or as directed by RML senior staff.

3.1.5 Close the detector shield.

3.1.6 Start count of the energy calibration thorium source for 10 minutes live time (600 sec.).

NOTE 1: *The thorium energy calibration data is stored automatically in a computer report file. This computer report file is used to document the calibration and the control status for each Ge spectrometer system.*

NOTE 2: *The RML computer system analyzes the thorium spectrum, computes the energy and width equations, and saves the spectrum along with the energy calibration values in the detector analysis slot. This calibration data will also be stored with each spectrum for subsequent analysis. The RML computer system makes it possible to retain the thorium energy calibration block with each subsequent analysis spectrum on that particular detector. Information associated with the energy calibration is automatically recorded in the computer database. The computer also performs QC checks and will warn the operator if the current calibration is outside of established criteria.*

3.1.7 When count if finished, run the energy calibration analysis routine (energy scale calibrate).

3.1.8 Ensure the detector slot, thorium source number, sequence number, efficiency table, and distance is entered for the program being run.

3.1.9 Remove the source from the source card sample holder.

3.1.10 Examine the printed copy of the energy calibration information for acceptable values.

3.2 Low-Energy Photon Spectrometer (LEPS) Energy Calibration

NOTE: *Energy Calibration is not required on detectors that are out-of-service (OOS).*

3.2.1 Perform an LEPS detector energy calibration per this section based on the following criteria:

- If a QA check (section 3.4) is out-of-specification.
- An energy calibration may be performed anytime deemed necessary by the lab analyst.

WARNING

When handling sources, failure to minimize handling time, use shielding, or use remote handling devices such as handles or tongs may result in increased personnel exposure. [JSA]

NOTE: *LEPS systems are normally used to measure gamma rays and x-rays over the energy range from a few keV (1 to 5) to a few hundred keV (600 to 800). Because of this low-energy range, a thorium source is not recommended for energy calibration. Other sources with gamma rays (not x-rays) in this energy range (such as Co-57, Sb-125, I-129, Ba-133, Eu-155, or Am-241) are used for energy calibration of LEPS systems. If x-rays are being measured for quantification, an appropriate x-ray emitting radionuclide source is used. The RML LEPS system is normally operated with an energy per channel (8192) of about 0.1 keV.*

3.2.2 Open the detector shield (iron room door).

3.2.3 Ensure only the source(s) to be used for this procedure will be in use in the area.

3.2.4 Place an energy calibration source (or sources) near the LEPS detector.

3.2.5 Adjust the source-to-detector distance to achieve approximately 2,000 counts per second (cps).

3.2.6 Close the detector shield (iron room door).

3.2.7 Count the energy calibration source(s) long enough to obtain photopeaks with counting statistics that produce well-defined photopeak centroids (normally a few thousand counts in the peak channels).

3.2.7.1 Obtain photopeaks at several well-distributed locations over the spectrum range.

3.2.8 Analyze the spectrum or spectra with a gamma-ray analysis program that determines the photopeak locations the same way as the sample analysis programs.

3.2.9 Make a record of the photopeak centroids and their known energies.

3.2.10 If the photopeaks are not identified in the known locations, then run the manual input program by inputting values for the measured peak channel locations and their associated known energies.

3.2.11 Review and verify the printed results.

3.3 Background Check Procedure

3.3.1 Close the detector shield.

NOTE: *Normally routine backgrounds have a count duration of 12 hours. However, RML senior staff may recommend a background count be a duration other than 12 hours.*

3.3.2 Start background check count on the detector (normally 43200 sec.).

3.3.3 Record count information on the RML sample log sheet

3.3.4 When the count is finished (normally after 12 hours), save data on disk on RML computer system.

3.3.5 Analyze the background spectrum.

3.3.6 Compare background results to previous results by control charting.

3.4 QUALITY ASSURANCE (QA)

NOTE: *QA check is not required on detectors that are out-of-service (OOS).*

3.4.1 Perform a QA check per this section based on the following criteria:

- For Ge or Ge(Li) detectors, a QA check is performed each calendar month.
- For LEPS detectors, a QA check is performed prior to use.
- Anytime a QA check is deemed necessary by the lab analyst.

WARNING

When handling sources, failure to minimize handling time, use shielding, or use remote handling devices such as handles or tongs may result in increased personnel exposure. [JSA]

3.4.2 Place the appropriate source(s) in the source holder:

3.4.2.1 If checking a Ge detector, place the Eu-152 source in the source holder.

3.4.2.2 If checking a LEPS detector, place the appropriate source(s) (Co-57, Sb-125, I-129, Ba-133, Eu-155, Am-241, or an appropriate x-ray emitting source) in the source holder.

3.4.3 Adjust the source holder to the proper source-to-detector distance:

NOTE: *The B7 (RJG-3) detector system has a source-to-detector distance of 0 cm. RML senior staff may recommend other source-to-detector distances (for other detector systems) for QA check at their discretion.*

3.4.3.1 If using the B7 (RJG-3) detector system, adjust source-to-detector distance to 0 cm.

3.4.3.2 If using a detector system other than B7 (RJG-3), adjust source-to-detector distance to 10 cm or as directed by RML senior staff.

3.4.4 Close the detector shield.

3.4.5 Record information on the RML sample log sheet.

3.4.6 Start QA check count on the detector (normally 1800 sec.).

3.4.7 When count is finished, save data on disk on RML computer system.

3.4.8 Analyze and print the QA check source spectrum with the analysis program ensuring results are in disintegrations per second (dps).

3.4.9 If the results are not automatically charted by the program, chart results in the lab QA logbook.

3.4.10 Report out-of-specification results to RML senior staff.

4. RECORDS

The records listed below are generated by the Analytical Laboratories Department and are managed in MCP-2007, Analytical Records Management.

Records Description	Uniform File Code	Disposition AuthorityRetention Period
Raw data	**7101**	***See, MCP-2007, Analytical Records Management***
RML sample log sheet	**7101**	***See, MCP-2007, Analytical Records Management***
RML QA logbook	**7101**	***See, MCP-2007, Analytical Records Management***
Calibration/background summary report	**7101**	***See, MCP-2007, Analytical Records Management***

5. REFERENCES

5.1 JSA ACLP-10.41

5.2 MCP-2007, Analytical Records Management

5.3 MCP-2391, Calibration Program

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

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

5.6 MCP-3635, Chemical Hygiene Plan

5.7 PLN-153, Quality Assurance Project Plan for the Analytical Laboratories Department Radioanalytical Section

6. APPROVALS

POSITION TITLE	SIGNATURE	DATE
Method Author		3-25-03
Responsible ALD Tech Leader		3-25-03
Responsible ALD Supervisor		3/25/03
ALD QA Officer		04/18/03
ALD Manager		4/1/03
ALD Facility Manager		3/27/03

Appendix A
Procedure Basis

Step/Section	Basis/Summary	Source
All	Establish calibration and calibration check procedure for M&TE used in laboratory.	MCP-2391
2.5, 2.6 A, 2.6 B, 4.1.2, and all Warnings	Controls are implemented to adequately mitigate hazards identified at JSA walkdown.	JSA ACLP-10.41
2.2	Use of blank samples are defined in PLN-153.	PLN-153, section 9.

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