

CP1

GAMMA SPECTROSCOPY

PART A

INTRODUCTION

The basic gamma ray spectroscopy system (references 1 and 2) consists of the dewar, shield, detector, preamplifier, amplifier, high voltage supply, analog to digital convertor, multichannel analyzer, memory disc drives, data analyzer, and a printout device.

Initial setup of new equipment should be done by the vendor. New operators are trained by experienced laboratory personnel. After learning the fundamentals, reference 3 should be consulted for additional details. Particularly difficult problems should be discussed with a system analyst or the vendor.

PRINCIPLE

Gamma emitting samples are placed on or near well shielded solid state gamma detectors which have been calibrated for several useful geometries. A linear energy spectrum is acquired and stored in memory. The spectrum is analyzed by a computer which provides a print out of peaks, energies, counts, nuclides, activity, minimum detectable activities, and the associated two sigma errors.

REFERENCES

1. American National Standard, ANSI N42.14, 1991, Calibration and Usage of Germanium Detectors for Measurement of Gamma Ray Emission of Radionuclides.
2. Dale W. Nix, Robert P. Powers and Larry G. Kanipe, Application of Germanium Detectors to Environmental Monitoring, EPA_600/7_79_054, March 1979.
3. Instruction Manual for Canberra Apex Gamma software, V1.2.
4. Annual Book of ASTM, Standards Vol. 11.02, pp. 310-320.
5. National Nuclear Data Center at Brookhaven National Laboratory
www.nndc.bnl.gov/
6. The Berkley Laboratory Isotopes Project's, Richard B. Firestone
<http://ie.lbl.gov/education/isotopes.htm>

Certification Record for

CP1

GAMMA SPECTROSCOPY

CHECKPOINTS

- 1. **JOB HAZARD ANALYSIS (JHA)** _____
- 2. **MSDS/HAZARDS DISCUSSED** _____
- 3. **LINEARITY CHECK** _____
- 4. **SAMPLE SET-UP** _____
- 5. **DATA ACQUISTION** _____
- 6. **QC RESULTS** _____

ANALYST'S SIGNATURE: _____

CERTIFIED BY: _____

DATE: _____

COMMENTS: _____

PART B

1.0 PURPOSE AND SCOPE

This section provides an outline of the procedure for acquiring and analyzing gamma spectra. This should be combined with the Canberra Operating Manuals and hands-on training for details.

2.0 APPARATUS

Dell Precision 690 PC or equivalent
Marinelli beakers and other appropriate containers
NIST traceable standards or industry accepted reference material
Solid state gamma detectors

3.0 PROCEDURE

Before proceeding, you must be certified as indicated in QCP1 of this manual and Section 3 of the Quality Program (QP) Manual. Quality control requirements for this procedure are defined in QCP5. These requirements may be augmented by requirements in a client's statement of work. See page two for a copy of the certification record. Review the JHA for IEAV Count Room Operations.

3.1 Startup Operation

3.1.1 Select Apex Gamma Icon on the Desktop.

3.2 Daily Quality Control Counting and Acceptance Criteria

3.2.1 Place QC sample designated for each detector on the detector.

3.2.2 Select **Main** from the Apex View tool bar.

3.2.3 Select **QA Check** for desired detector.

3.2.4 Select **Calibration Check** in the window.

3.2.5 Repeat steps 3.2.3 and 3.2.4 until the QA is running on all detectors.

3.2.6 A report providing all necessary information for the QC review will be generated at the end of the count.

- 3.2.7 The daily QC review sheet for each detector indicates six parameters that have to pass before the detector can be used to count samples. They are:

Decay Corrected Activity (DCA) peak 351
DCA peak 609
DCA peak 1764
Full Width at Half Maximum (FWHM) peak 351
FWHM peak 609
FWHM peak 1764

If no “ACTION” flags are indicated on the report for these parameters, go to step 3.2.10

NOTE: The parameters are set up initially based on 30 counts of each QC sample. The Apex Gamma software automatically calculates the limits that establish the acceptance criteria. Investigation limits are between ± 2 sigma and ± 3 sigma. The action limit is greater than ± 3 sigma.

- 3.2.8 When an “ACTION” flag is generated, the daily QC sample must be run two more times and all parameters must pass both times for the detector to be used to count samples.
- 3.2.9 If an “ACTION” flag is generated for any of the six parameters on either of the next two QC counts, the detector is taken out of service until the problem is resolved.
- 3.2.10 The operator will initial and date the daily QC review sheet for each detector whether or not samples are to be counted on the detector.

3.3 Sample Counting

- 3.3.1 Select **Sample** from Apex View tool bar.
- 3.3.2 Select the **Samples** tab from sample view.
- 3.3.3 Select sample type. All samples are defined as “Gamma”.
- 3.3.4 Input Sample ID.
- 3.3.5 Select the Procedure to be used. The Geometry, count time, library, analysis sequence file and report file will be displayed. Click **Next**.
- 3.3.6 Input sample date, time, weight and units. Click **Save**.
- 3.3.7 Place the sample on the detector to be used.

- 3.3.8 Select Main from the Apex Views tool bar.
- 3.3.9 Drag the sample from the available list to the detector on which the sample has been placed.
- 3.3.10 The gamma spectroscopy data is imported into the program database using the **TOOLS** tab in the database. If the database is not functioning, a printed report will be generated and maintained as part of the task record.

4.0 DATA REVIEW

- 4.1 Once the sample has completed counting, the sample data are stored under a unique file configuration. This configuration contains the sample information as well as counting results and must be reviewed for accuracy. Items to be reviewed on the printout or in the database are as follows:

- Sample ID
- Sample weight
- Sample geometry
- Sample date
- Sample matrix
- Peak resolution (FWHM in keV)
- Linearity
- Isotopic interferences
- Unrequested isotopes

- 4.2 The operator and the Laboratory Manager or designee is responsible for reviewing the gamma spectroscopy data. If there is a problem, the problem is resolved, the data are regenerated, and the review process starts over.
- 4.3 After the reviews are completed, the data are transferred to the appropriate task data folder, either hardcopy and/or electronic, and also to the person responsible for the project.

5.0 CALCULATIONS

- 5.1 Gamma spectra are analyzed by the Dell Precision 690 using software provided by Canberra. The results are given for peak search, peak identification, quantities of nuclides, counting errors, rejected and unknown peaks, and minimum detectable activities.
- 5.2 Critical data values are documented in the electronic configuration file on each sample. The following equations define the critical data values.

$$Concentration = \frac{G - B}{E \cdot Q \cdot AB \cdot T \cdot 2.22} = pCi / unit$$

$$2\sigma \text{ Error} = \frac{1.96\sqrt{(G+B)}}{E \cdot Q \cdot AB \cdot T \cdot 2.22} = \text{pCi/unit}$$

In the event of G=0 and B=0, 1 will be substituted for G+B to prevent a division by or into zero error in this uncertainty calculation.

$$2\sigma \text{ TPU} = C \cdot 1.96 \sqrt{\frac{(G+B)}{(G-B)^2} + (RE)^2 + (RAB)^2 + (RQ)^2} = \text{pCi/unit}$$

In the event of G=0 and B=0, 1 will be substituted for G+B/(G-B)² to prevent a division by or into zero error in this uncertainty calculation.

$$\text{MDC} = \frac{3 + 4.65\sqrt{B}}{E \cdot Q \cdot T \cdot AB \cdot 2.22} = \text{pCi/unit}$$

where:	AB	=	gammas/disintegration
	B	=	background counts
	C	=	concentration
	E	=	counting efficiency counts/gamma
	G	=	sample gross counts
	MDC	=	minimum detectable concentration
	Q	=	quantity
	RAB	=	relative percent error of the gamma abundance
	RE	=	relative percent error of the counting efficiency
	RQ	=	relative percent error of the quantity
	T	=	time in minutes
	TPU	=	total propagated uncertainty
	2.22	=	dpm/pCi conversion

6.0 CALIBRATION

6.1 Calibration Standard Preparation

- 6.1.1 Select a NIST traceable mixed gamma standard. For water samples, prepare the appropriate volume of carrier solution by adding approximately 100 mg of nonradioactive carrier for each nuclide in the calibration standard. Weigh a portion of the standard that will give approximately 1E4 pCi per isotope and add to the appropriate volume of carrier solution (0.5 or 1 liter). Stir overnight, then transfer the solution to the counting container and submit to the counting room.

- 6.1.2 For soil standards, weigh an amount of standard that will give a range of 3 pCi/g to 50 pCi/g per isotope. After adding the standard to the carrier solution and mixing, add the solution to the chosen calibration matrix. It is preferable to make up one large standard soil from which all counting geometries can be taken, but it is permissible to make up standards for each counting geometry. Because of density considerations, soil standards are made using top soil and sand. The soil standards have to be weighed before the addition of the liquid standard and after the soil standard has completely dried. The top soil contains naturally occurring radioactivity, but this activity is insignificant when compared to the standard.
 - 6.1.3 The standard soil/sand is blended in a mechanical shaker for one hour or on a ball mill overnight. Place the standard in the proper counting geometry container and submit to the counting room.
 - 6.1.4 For air filters, coat one side of a 2 inch filter with clear enamel and allow the paint to dry. Add a weighed amount of standard to the uncoated side that will give approximately 1E3 pCi/filter per isotope to the filter. Distribute the standard as evenly as possible around the filter. Allow the filter to dry completely. Place the filter in a Petri dish or a plastic zip-lock bag and submit to the counting room.
- 6.2 Calibration Certificate Files
- 6.2.1 Select **Setup** on the Apex view tool bar.
 - 6.2.2 Select the **Editors tab** in the Setup view.
 - 6.2.3 Select the **Certificate** button.
 - 6.2.4 Select **File New**.
 - 6.2.5 Input Certificate file name.
 - 6.2.6 Input the weight, in grams, of the calibration standard added to the new geometry.
 - 6.2.7 Input the calibration standard identification, e.g., QCY48 R4/285/68.
 - 6.2.8 Input the reference data of the calibration standard.
 - 6.2.9 Input the activity, in gammas/second/gram, for each radionuclide.
 - 6.2.10 Input the uncertainty, in percent, for each radionuclide.
 - 6.2.11 After reviewing the new certificate values, print the certificate file.

- 6.2.12 Each printed certificate file is to be signed and dated by the person creating the file.
- 6.2.13 The Laboratory Manager or designee will review and approve each certificate file before step 6.3 is started. Approval will be documented by signing and dating the certificate file.
- 6.3 Efficiency and Energy Sample Count
 - 6.3.1 Select **Calibration** from the Apex View tool bar.
 - 6.3.2 Select **Count** Tab in the calibration view.
 - 6.3.3 Choose Efficiency or Energy in the Count Type window.
 - 6.3.4 Select the detector to be calibrated from the pull down menu.
 - 6.3.5 Select **Geometry** from the pull down menu.
 - 6.3.6 Select the **Certificate** from the pull down menu.
 - 6.3.7 Place standard on the detector, clear the screen and start the count.
- 6.4 Efficiency Calibration
 - 6.4.1 Select **Calibration** from the Apex View toolbar.
 - 6.4.2 Select the **Efficiency** tab in the Calibration view.
 - 6.4.3 Select the **Detector** from the pull down menu.
 - 6.4.4 Select the **Geometry** from the pull down menu.
 - 6.4.5 Choose **Calibration** count in the create calibration window. Click Next.
 - 6.4.6 Select count to be used.
 - 6.4.7 The curve will be shown. Click **View Report**, then print.
 - 6.4.8 Save the calibration.

6.5 Approve Efficiency

6.5.1 Select **Calibration** from the Apex View toolbar.

6.5.2 Select the **Efficiency Approve** tab.

6.5.3 Select the Detector and Geometry.

6.5.4 Select the calibration count to be approved. Click **Next**.

6.5.5 Click the **Approve** button.

7.0 RECORDS

7.1 Reference QP Manual for general record requirements.

7.2 A full system backup is performed daily by ISD to protect spectra, data and system files that were generated during the previous day.

7.3 There are no hard copy assignment, data, or concentration and uncertainties sheets generated for this process.

7.4 Calibration information is maintained electronically.