CP1

GAMMA SPECTROSCOPY

PART A

INTRODUCTION

The basic gamma ray spectroscopy system (references 1and 2) consists of the dewar, shield, detector, preamplifier, amplifier, high voltage supply, analog 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 should be 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 Procount software.
4. Instruction Manual for Canberra VMS Personal Workstation.
5. Annual Book of ASTM, Standards Vol. 11.02, pp. 310-320.

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 protocol sheets and hands-on training for details.

2.0 APPARATUS

VMS Personal Workstation
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 QA Manual. See page two for a copy of the certification record. Review the JHA for ESSAP Count Room Operations.

3.1 Startup Operation

3.1.1 The user name for Procount operations is **GAMMA**.

3.1.2 On the Sessions Manager window select **Applications**.

3.1.3 On the Applications pull down menu select **Procount GUI**.

3.1.4 On the Spec Assistant menu select **File**.

3.1.5 On the File pull down menu select **Open Work Space**.

3.1.6 Open the **Gamma.WSP**.

3.1.7 Procount window will be displayed.

3.2. Daily Quality Control Counting and Acceptance Criteria

3.2.1 Select **QC** from the Procount menu.

3.2.2 Select **Calibration Check** from the **QC** pull down menu.

3.2.3 Select Detector to be used.

3.2.4 Select Geometry to be used: Detector 6-**LM**, detector 7-**FM**, or detector 8-**LM.**

3.2.5 Select certificate to be used. Example: **Daily QA DET6** for detector 6.

3.2.6 A report will be generated at the end of the count.

3.2.7 The daily QC review sheet for each detector will have 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 there is not an action flag, go to step 3.1.10

NOTE: The parameters are set-up initially based on 30 counts of each QC sample. The Procount 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 sign and date the daily QC sheet for each detector whether or not samples are to be counted on the detector.

3.3 Sample Counting

3.3.1 Select **Count** from the main Procount menu.

3.3.2 Select **Start a Count** from the **Count** pull down menu.

3.3.3 Select the detector to be used for the count.
NOTE: ***Acquisition automatically starts after this step.***

3.3.4 Select the analysis Sequence file.

3.3.5 Select the geometry.

3.3.6 Input sample information, including the sample number, quantity, date, and analyst.

3.3.7 Place the sample on the detector to be used.

3.3.8 Clear the MCA with the **Clear** button on the MCA screen.

3.3.9 A report will be generated at the completion of the count.

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 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 and also to the person responsible for the project.

5.0 CALCULATIONS

5.1 Gamma spectra are analyzed by the VMS Personal Workstation 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.

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

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

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

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

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

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/ml of nonradioactive carrier for each nuclide in the calibration standard. Weigh a portion of the standard that will give approximately 1E4 pCi/g 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 Standard Count

6.2.1 Select **Calibration** from the main Procount menu.

6.2.2 Select **Count Calibration Spectrum** from the **Calibration** pull down menu.

6.2.3 Select the detector to be calibrated.

6.2.4 Input desired counting time.

6.3 Efficiency Calibration

6.3.1 Select **Efficiency Calibration** from the **Calibration** pull down menu.

6.3.2 Select the detector to be calibrated.

6.3.3 Select the geometry or C**reate New Geometry** and type the new geometry name.

6.3.4 Select the certificate for the standard counted.

6.3.5 The efficiency calibration curve will appear.

6.3.6 Select fit **EMP**.

6.3.7 Select scaling linear and select **OK**.

6.3.8 The calibration report will come to the screen. Select print for reviewing and filing.

6.3.9 Select **OK** to store calibration results.

6.3.10 Select **Yes** to save geometry.

6.4 Approve Efficiency

6.4.1 Select **Approve Efficiency Results** from the **Calibration** pull down menu.

6.4.2 Select the Geometry for the efficiency requiring approval and select **OK**. For new efficiencies, a known sample that is different from the standard must be analyzed and the measured to known values must be statistically equal to 1.0. For updating efficiencies, a comparison of efficiencies must agree within 10 percent. The Laboratory Manager must approve the final efficiency curve.

6.5 Store Calibration Coefficients

6.5.1 Select **Store Calibration Coefficients** from the **Calibration** pull down menu.

6.5.2 There are three "storing" options available. Select **Store Efficiency Coefficients.**

6.5.3 Select the detector that has been calibrated.

6.5.4 Select the geometry that was calibrated.

7.0 RECORDS

7.1 Reference QA Manual for general record requirements.

7.2 A system backup to DAT tape or equivalent is performed weekly to protect spectra and data that were generated during the previous week.

7.3 A full system backup to DAT tape or equivalent is performed monthly. This backup includes system operating files and all files located on the hard drive.

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

7.5 Calibration information is maintained electronically.