CABRERA SERVICES
RADIOLOGICAL · ENGINEERING · REMEDIATION

OPERATING PROCEDURE

FOR

GAMMA SPECTROSCOPY OPERATIONS
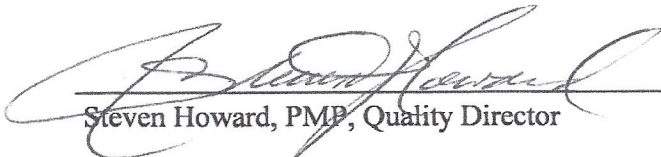
OP-029

REV. 3

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_____   __4/28/2011__
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1.0 GAMMA SPECTROSCOPY OPERATIONS

2.0 APPLICABLE MATRIX

This section is not applicable to this specific operating procedure.

3.0 DETECTION LIMIT

This section is not applicable to this specific operating procedure as this procedure presents basic operation of equipment. Project specific work plans and sample analysis methods should be referred to for specific required detection limits using this equipment.

4.0 SCOPE AND APPLICATION

This operating procedure provides instruction and requirements for the operation of CABRERA SERVICES, Inc. (CABRERA) gamma spectroscopy systems. Instructions are provided for proper equipment setup, routine instrument operation, quality control measurements, in-field container component counting, geometry modeling, and spectrum file management.

5.0 SUMMARY OF METHOD

This procedure describes the use of the Gamma Acquisition and Analysis (GAA) application within standard Canberra Industries' Genie-2000 software and general methods of performing gamma spectroscopy measurements. This operating procedure has been revised to meet the requirements of the Department of Defense Quality Systems Manual (DoD, 2003) and to incorporate both laboratory and non-destructive assay operations from CABRERA'S former procedure, Gamma Spectroscopy Laboratory Operational Procedures, OP-029, Rev. 3.

6.0 DEFINITIONS/ACRONYMS

6.1   FWHM – Full Width at Half Maximum

6.2   GAA – Gamma Acquisition and Analysis

6.3   HPGe – High Purity Germanium

6.4   ISOCS – In-situ Object Counting System

6.5 LN_2 – Liquid Nitrogen

6.6   MCA – Multi-Channel Analyzer

6.7   NIST – National Institute of Standards and Technology

6.8   SME – Subject Matter Expert

7.0 INTERFERENCES

This section is not applicable to this specific operating procedure.

8.0 SAFETY

8.1   Personnel handling radioactive material are trained in accordance with Cabrera's administrative procedure, AP-009 *Training*, and appropriate precautions will be adhered to.

8.2   Appropriate As Low As Reasonably Achievable (ALARA) measures shall be used to minimize the potential for contamination and unnecessary dose when in the presence of radiological material.

8.3   The detector systems utilize a high voltage. Use caution when handling cables and other electrically powered elements of the systems; ensure elements are powered down before unplugging.

8.4   Avoid skin contact with liquid nitrogen (LN_2) to avoid frostbite. When handling LN_2, always use proper eye and skin protection, including cryogenic safety gloves, safety glasses and/or a face shield.

8.5   The shielding for the detectors are comprised of painted lead. If the shielding gets scratched or cracked, alert the Safety Officer and take the necessary precautions.

8.6   The detector systems can be extremely heavy. Take care to watch for pinch points and when lifting or moving parts of these systems.

9.0 EQUIPMENT AND SUPPLIES

9.1   Liquid nitrogen
- Tank containing low (~22) Pounds per Square Inch (PSI) liquid nitrogen
- Transfer dewar (optional)
- Metal connection hose
- Rubber tubing with clamps (optional)
- Adjustable Crescent Wrench (~12")
- Face mask, apron, cryogenic gloves

9.2   Detector
- Dewar (for lab systems)
- Composite cable
- Detector Specification Sheet
- Allen wrench (7/32")

9.3   Multi-Channel Analyzer (MCA)
- DSA 1000 or InSpector 2000
- USB cable
- RS-232 serial cable (for analog MCA only)
- USB hardware key (for DSA-100 only)

9.4   Shield
- For lab units this will consist of a lead table and stackable rings with a sliding door on top.
- For In-Situ Object Counting Systems (ISOCS) this will consist of a shield set: (4) annular shield rings (25 mm or 50 mm), steel spacer tubes, clamp screws, and detector clamps.
- Standard/Philips screwdrivers and standard/SI Allen wrench sets.

9.5   Uninterruptable power supply and conditioner and a digital voltmeter

9.6   Computer with GENIE-2000 and ISOCS Geometry Composer software installed, printer, USB flash drive, extension cords with surge protectors, a printer with necessary accessories and a bound logbook.

9.7   ISOCS systems may also require the use of the following equipment (actual list will vary depending upon need):
- Detector Tripod
- Detector Cart (hydraulic lift or Canberra standard)
- Extra long Signal Cable
- 12V car battery with A/C inverter
- Source jig for daily QC measurements

10.0   REAGENTS AND STANDARDS

This section is not applicable to this specific operating procedure.

11.0   SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

This operating procedure applies strictly to the operation of the gamma spectroscopy counting systems; therefore this section is not applicable.

12.0   QUALITY CONTROL

12.1   Data collected with any high-purity germanium (HPGe) detector is considered invalid unless compliance with the quality assurance requirements stated herein is demonstrated and documented.

12.2   Individuals performing measurements with CABRERA Gamma Spectroscopy

equipment shall be familiar with the requirements established in the current and approved version of this procedure, shall have a general knowledge of gamma spectroscopy principles and shall have successfully demonstrated operation and setup of the equipment.

12.3   The instruments are susceptible to water/moisture damage and shall only be operated in wet environments when appropriate protective precautions are implemented. The instruments should not be operated in very wet environments such as heavy precipitation.

12.4   The instrument shall not be operated in environments where the ambient temperature exceeds 50° C or is less than -10° C (122° F and 14° F, respectively).

12.5   Daily quality control checks shall be conducted each day the systems are used. Please see Section 14.4 of this operating procedure for further details regarding daily control checks.

12.6   Quality control samples will be performed at the frequency prescribed in the project work plans, e.g., 10% means a QC count (lab duplicate, field duplicate, replicate, blank, etc.) will be performed on every tenth sample.

12.7   If QC control samples do not pass the QC parameters stated in the project specific work plans or in the sample analysis procedure consult the Project HP or Lab Manager for further guidance.

12.8   Laboratory replicate sample measurements are performed to test the reproducibility of the HPGe system. They should be near identical counts of a single sample with all system parameters remaining constant between the two.

12.9   Samples flagged to be replicates shall be marked with a "R" on the label and set aside for counting. Replicates may be counted immediately or the next business day. Replicates MAY NOT be compiled for counting all on the same day.

12.10   A method blank is a "zero-activity" sample run through the entire sample preparation and counting process to ensure that no biases exist in the laboratory and to ensure that the detector and/or shield interior has not become contaminated inadvertently.

13.0   CALIBRATION AND STANDARDIZATION

The gamma spectroscopy detectors shall be calibrated upon setup. Calibrations and/or gain adjustments shall be performed when necessary as stated herein to ensure that the accuracy of measurement results is not compromised. Please see Section 14.2.12 of this procedure for specific details regarding this process.

14.0   PROCEDURE

14.1 GAA Screen and Fields

The Acquisition and Analysis screen is shown in Figure 1. Instrument controls and functions are performed via this screen. The menu tool bar is the second line of the screen and begins with the word File. Each of these words or menus has submenus, which can be obtained by single-clicking one of the main menus from the toolbar. Directly below the menu bar is a shortcut bar or toolbar, which eliminates the need for scrolling through the menu bar for frequently used tasks. The top portion of the spectrum screen displays the spectral data and information about the spectrum. The very bottom of the screen displays the generated report of the spectrum upon analysis. Any page of the report can be viewed by scrolling.
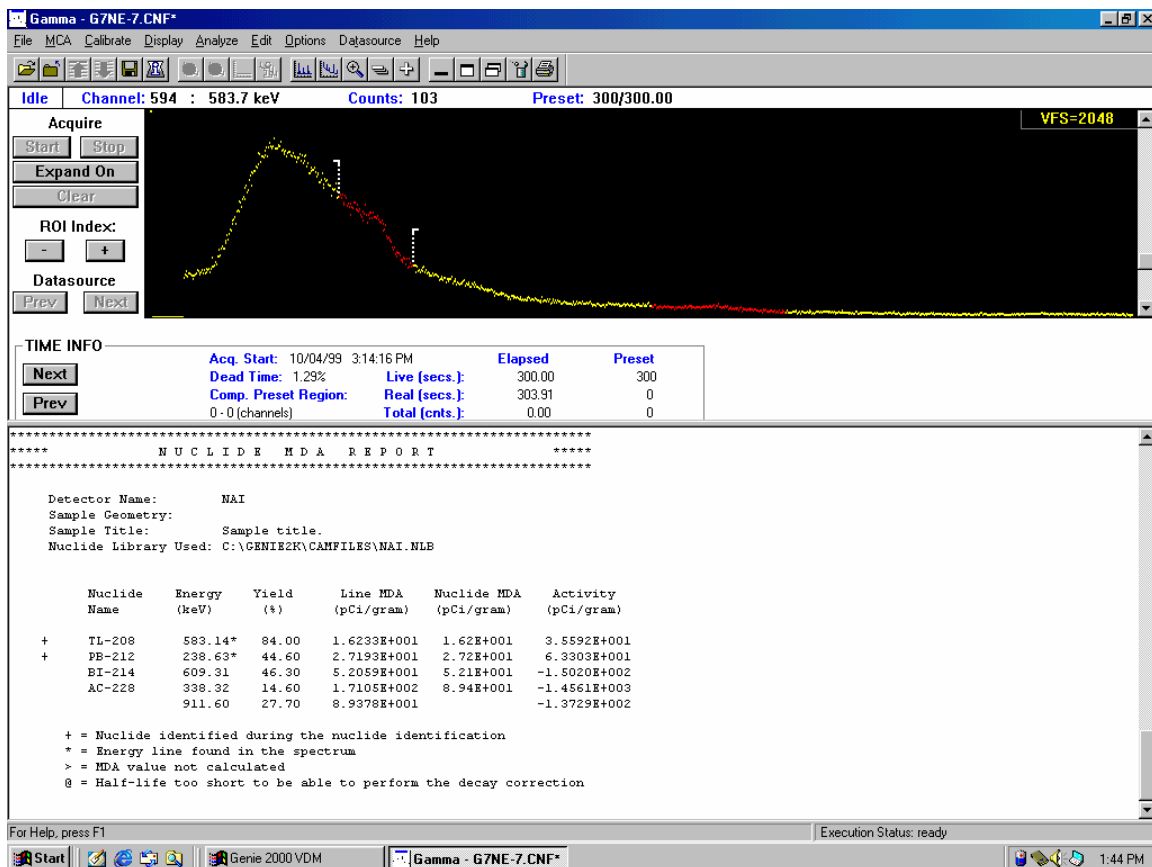

Figure 1. GAA Screen with Analysis

14.2 Basic Instrument Operation

14.2.1 Launching GAA

GAA can be opened from the Windows desktop by double-clicking on the GAA Icon or by choosing **Start**, then **Programs**, then **Genie-2000**, then GAA. Before opening the detector, the MCA must first be switched on. The switch is located on the left side of the InSpector, and in the back of the DSA.

14.2.2 Opening the Detector

Choose **File** (located on the menu bar), then **Open Datasource.** Open source type **Detector** and select the specific detector (e.g. DET01).

14.2.3 Applying High Voltage to the Detector

Caution: <u>Ensure that the detector has been properly cooled with liquid nitrogen (i.e. for approximately six hours) before performing this step</u>

Choose **MCA** (located on the menu bar), then **Adjust**. A box will appear in the lower portion of the screen. Select the **HVPS** tab. Select the **On** button, after the **Wait** signal has disappeared from the top left corner of the Adjust box, and exit from the **Adjust** box by single-clicking on the **Exit** button.

14.2.4 Selecting the Appropriate Time

Choosing the appropriate count time can be accomplished by using the edit icon located on the shortcut menu bar or by selecting **MCA,** then **Acquire Setup** from the menu bar. Enter the appropriate time where prompted. Count time <u>must</u> be entered as **live time** for the instrument dead time correction to function properly. Refer to the project-specific QAPP or the SME for appropriate live time values.

14.2.5 Starting/Stopping Data Acquisition

In the main GAA screen, pressing the **Start** key under Acquire will cause data acquisition to begin. The **Start** button will no longer be bold face when the acquisition begins. Acquisition can be stopped at any time by pressing the **Stop** button located immediately to the right of the start button. If acquisition is stopped and a preset time is entered (see Section 14.2.4), pressing the **Start** button will resume acquisition, which will continue until the preset time is reached.

14.2.6 Dead Time Check

In the **Time Info** section of the GAA display window can be found the **Dead Time**. If the dead time increases to above 5% when no known radiological source is in the area or increases above 15% when a known radiological source is being measured, then contact the SME for evaluation.

14.2.7 Entering Sample Information

Descriptive information is entered to uniquely identify each acquired spectrum. This can be done while data acquisition is in progress, or after acquisition has stopped.

Click on **Edit** in the GAA window menu bar, then select **Sample info** from the **Edit** menu. This will display the Edit Sample Information dialog box, which includes descriptive fields for: Sample Title, Collector Name, Sample Description, Sample ID, Type, Units, and Sample Geometry.
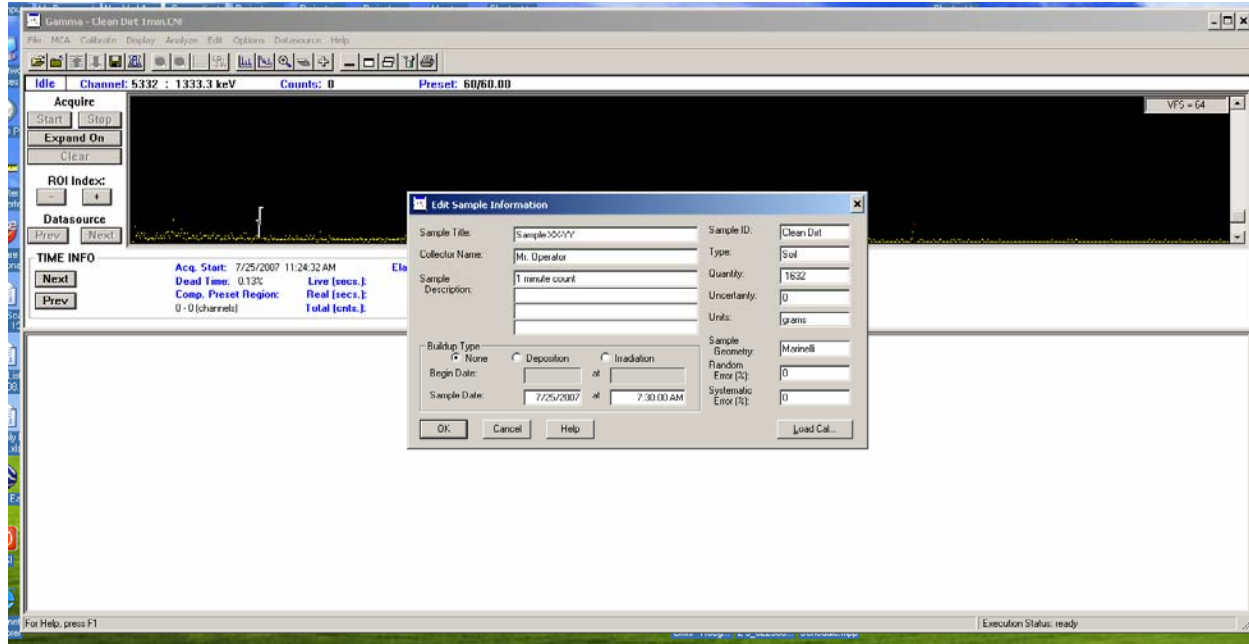
Figure 2. View of Edit Sample Information Dialog Box

Editable fields are also provided for Quantity, Uncertainty, Random Error, and Systematic Error. Select the 'None' button in the Buildup Type section. If it is necessary to ensure that short-lived nuclides are appropriately decay-corrected, then you can enter the date the sample was collected in the 'Sample Date' field.

Make all desired entries in the fields of the Edit Sample Information dialog box, then click on the **OK** button to save the information and exit this dialog box.

14.2.8 Clearing Spectrum Data

Pressing the **Clear** key under the **Start** and **Stop** buttons under Acquire will erase the spectrum data. Remember to save current detector settings by choosing the save disk icon on the tool bar before clearing data.

14.2.9 Information from the Spectrum Display

In the Spectrum Display, the left and right arrow keys will move the spectrum cursor. The cursor can also be moved via the mouse by single-clicking in the area of interest or clicking and dragging with the mouse. The information bar above the Spectrum Display will indicate the energy level (keV) and channel number corresponding to the location of the cursor, as well as the total number of counts (cts) at that energy level, and the Preset/Elapsed time. In Figure 1, the acquisition was completed, therefore, the elapsed time was 300 seconds and the preset time was 300 seconds (300/300 is shown in Figure 1). If the instrument had only reached 200 seconds of the preset 300 seconds, then the display would show 300/200.

The default settings for the GAA display allow the spectrum display vertical

scale to automatically adjust when counts in any channel exceed the current vertical scale. The initial vertical scale is 64. The spectrum Vertical Full Scale (VFS) display scale can be expanded or contracted by pressing the up and down arrow, respectively.

14.2.10 Creating/Deleting Regions of Interest

While in the Spectrum Display, a temporary ROI is established by using the **Control** and **L** keys and the **Control** and **R** keys, to define the beginning point and the ending point of the ROI, respectively. Information on an ROI is obtained by pressing either the **Next** or **Prev** buttons until Marker Info is displayed, see Figure 3.

Integral is the total number of counts within the ROI. Area displays the net counts in a region. An ROI can be made permanent, by selecting the **Add ROI** menu item under the **DISPLAY** menu.
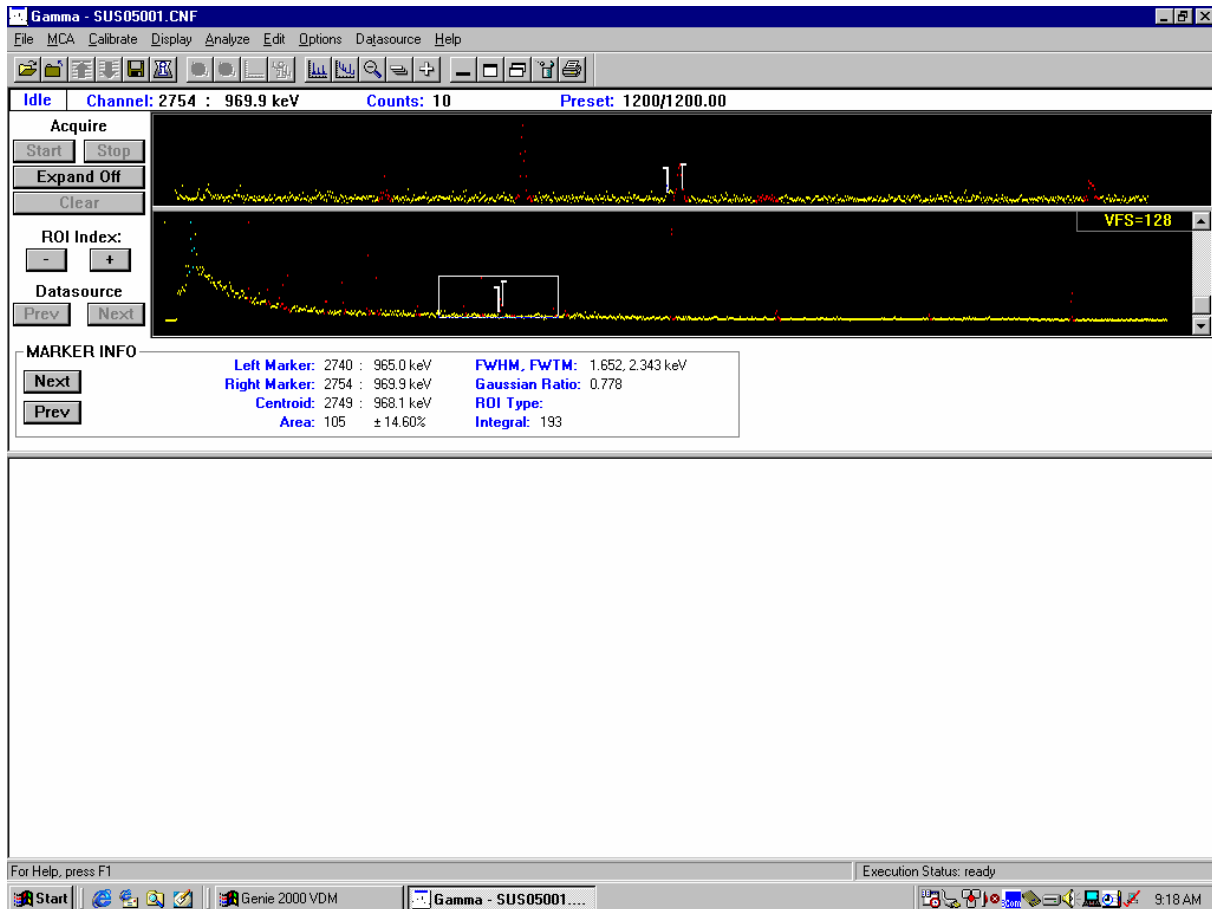

Figure 3. GAA Screen with Marker Info Displayed

14.2.11 Adjusting Amplifier Gain and ADC Zero

Prior to the start of each counting session the system alignment should be checked. If the energy gain or ADC zero is beyond acceptable limits, they must be adjusted.

To check and adjust the amplifier gain and ADC zero settings, a suitable check source should be placed near the detector during spectral data acquisition. This check source should emit photons with emission rates sufficient to produce readily detectable photopeaks. A NIST-traceable source may be used for this purpose but it is not mandatory.

The centroid channel and corresponding energy value for each reference photopeak should be compared to the expected values. Observed photopeak centroids should typically be within +/- 0.5 keV of the expected energy values. Adjust the vertical full scale of the displayed spectrum and use the **Expand On** button, as appropriate, to view the reference photopeaks.

From the MCA window, click **Acquire Start** and note the position of the photopeaks in the spectrum (specific to the source being used). If necessary, amplifier gain and/or ADC zero settings can be changed by selecting **Adjust** from the MCA menu. This will display the Adjust dialog box.

The scroll bars in the Amplifier '**Fine' gain, or Superfine 'S-fine' gain** sections of this dialog box should then be used to make the appropriate changes. After each change, clear any previous spectral data and start a new acquisition to evaluate the new settings.

When spectral data acquisition using a gamma source with known photopeak locations indicates that the centroid channel of each reference photopeak is within +/- 0.5 keV of the expected energy values, the new settings are deemed acceptable. Click on the **Exit** button to exit the Adjust dialog box.

To save the new parameter values, click on **File** in the GAA window menu bar, then select **Save** from the **File** menu.

14.2.12 Energy and Efficiency Calibrations

Detector calibrations will be performed as needed by, or with, the assistance of an individual qualified to perform detector calibrations.

14.2.13 Saving a Spectral Data File

After spectral acquisition has stopped, the spectral data and related information can be saved in a file. This step is required before analyses of the acquired spectrum can be performed.

Click on **File** in the GAA window menu bar, then select **Save as** from the File menu. This will display the Save As dialog box. Enter the desired name, up to eight characters which will be followed by the .CNF extension in the File name field.

You may also enter an appropriate supplemental text string (up to 32 characters) in the Description field. Enter text that will be helpful in selecting this file when attempting to identify it at some later time.

Click on the **OK** button to save the file and exit the Save As dialog box. The default directory used to store these files is GENIE2K\CAMFILES.

To collect additional spectra, clear existing spectral data from the graphics region of the GAA window.

14.2.14 Loading Calibration Parameters

Canberra's Geometry Composer and GAA Software must be used to generate an efficiency calibration file appropriate for the object being assayed. The desired calibration parameters can be loaded and saved with the spectral data. The spectral data file can then be analyzed to determine nuclide activity concentration.

From the GAA window menu, choose **Calibrate**. Select **Load** from the Calibrate menu. This will display the Load Calibration dialog box. The File Names section in this dialog box will contain a list of all calibration files currently stored in the directory specified by the environment variable CALFILES.

When the Load Calibration dialog box is first displayed, the boxes labeled Energy/Shape and Efficiency will contain a check mark, Leave these boxes checked.

14.2.15 Analyzing a Spectral Data File

After opening a CAM file datasource and loading the appropriate efficiency calibration parameters, spectral data analysis can be performed. The **Analyze** options in the GAA window menu bar can be used to perform these analyses.

Choose **Analyze** in the GAA window menu bar to display the Analyze menu. Next, choose **Execute Sequence** and select the sequence specified for what you are counting.

14.2.16 Exiting the GAA Application

When all spectral data analyses have been completed, click on **File** in the GAA window menu bar. Select **Close** from the File menu. If a datasource with unsaved changes was still open in the GAA window, a message

screen will be displayed asking whether the latest changes should be saved. Respond appropriately to any such message.

If you are shutting down the system, before completely exiting from GAA, turn off the high voltage to the detector by reversing the steps for applying high voltage in Section 14.2.3. The detector high voltage will remain on once the system has been setup for operations and is not typically shut down unless the system is scheduled for warm-up, transportation/movement throughout the site, and/or shipping, or an error occurs.

14.3 In-Situ Use and Generation of In-Situ Object Counting System Models

14.3.1 General Information

Several of CABRERA's HPGe detectors may be used for *in situ* gamma spectroscopy applications. *In situ* applications require use of portable detectors with multi-attitude, i.e. 'Big MAC' cryostats.

14.3.3 ISOCS Applications

Consult with a SME or the Gamma Spec Program Manager for proper setup and use of a portable HPGe detector.

14.3.4 Big MAC Cryostat and Dewars

The Big MAC dewer must be filled with LN_2 every 2-3 days. When filling a Big MAC cryostat with LN_2, be sure to follow the fill and vent instructions provided near the two vents. The position of the vent cap depends upon the vertical orientation of the cryostat (detector facing up / facing down). If the detector is horizontal, either port may be used for fill or vent, but once assigned, you should not alternate.

14.3.5 ISOCS Modeling

Spectra files collected with an ISOCS unit will require a source mode, and monte carlo efficiency file created by Canberra's Geometry Composer software. All ISOCS$^®$ source models will be created and/or reviewed by a qualified NDA SME prior to their use.

Documentation will be provided for all ISOCS$^®$ models which will state the particular source specifications, underlying assumptions, and design dimensions for each model.

14.3.6 Loading an ISOCS Calibration File

Follow the instructions in Section 14.2.14. However, for ISOCS use, the dialog box labeled "Energy/Shape" needs to be unchecked. This will allow new efficiency calibration parameters to be loaded without changing the original energy/shape calibration, which is appropriate for ISOCS use of the detector. Use the vertical scroll bar in the File Names section until the desired calibration file name is shown, then double click on that file name. This will exit the Load Calibration dialog box, and load the efficiency calibration parameters from the selected file into the open CAM file datasource.

14.4 Daily Quality Control

14.4.1 A Daily QC Calibration Check is performed with an operational check source at the start of each field measurement shift. The check source should

be placed at a reproducible position relative to the detector end-cap. A typical arrangement for a lab QC jig is an empty marinelli beaker with the sources attached and/or positioned with its interior in a constant geometry.

14.4.2 Save the QC file with the active date in the filename to the project QA folder and transfer the results into the project QAF file using the **Quality Assurance Editor** program. This step can also be automated by editing the QC analysis sequence if it will minimize transfer errors.

14.4.3 The specific QC parameters evaluated and the acceptable ranges of results are summarized in the following table:

QC Calibration Parameter Description	Acceptable Range of Results
Low energy photopeak energy	Within fixed energy limits[1]
High energy photopeak energy	Within fixed energy limits[1]
Low energy photopeak FWHM[2]	Mean +/- 2 std. deviations[3]
High energy photopeak FWHM[2]	Mean +/- 2 std. deviations[3]
Low energy decay corrected activity	Mean +/- 2 std. deviations[3]
High energy decay corrected activity	Mean +/- 2 std. deviations[3]

Notes:

1. Observed peak energies should typically be within $\pm$ 0.5 keV of theoretical (approx. $\pm$ 3 channel limits)

2. FWHM stands for Full Width at Half Maximum. It is the width of the energy peak, in keV, at one half of the peak amplitude, or height.

3. The mean and standard deviation values for these QC parameters are established by performing a set of 10 initial counts. Subsequent count results are then compared to the established "investigation level" range of [mean +/- 2 std. dev.] and "action level" range of [mean +/- 3 std. dev.]. The mean and standard deviation is updated after each transfer to the QAF file.

14.4.4 If all QC parameter results are within the acceptable ranges, the gamma spectroscopy system shall be approved for routine use during the remainder of that day. If any of the results are outside the applicable acceptable range, the QC Check count shall be repeated. If the results of the second count are within the acceptable ranges, the gamma spectroscopy system shall be approved for routine use. If the results of the second count are outside the acceptable ranges, the system shall be taken out of service until the problem can be resolved by the Gamma Spectroscopy Program Manager.

14.4.5 QC parameter results from these daily QC Check counts are stored in a QA file and can be tabulated and plotted for long-term trending. Updated tables and plots shall be printed (hard copy or electronic) on a weekly basis and retained in a readily-accessible file. The SME shall review the QC check data weekly for trending. These printouts are intended to provide a record of stable system performance, or alert the user to possible problems.

Unless this process is automated, the QAF file shall be saved after importing the day's QC information prior to closing or it will require transfer again.

15.0   CALCULATIONS

This section is not applicable to this specific operating procedure.

16.0   METHOD PERFORMANCE

Please refer to section 14.0 of this operating procedure for information pertaining

to this section.

17.0   POLLUTION PREVENTION

This section is not applicable to this specific operating procedure.

18.0   DATA ASSESSMENT AND QC ACCEPTANCE CRITERIA

Please see Sections 12.7 and 14.4 for further details.

19.0   CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

Please see Sections 12.7 and 14.4 for further details.

20.0   UNACCEPTABLE DATA CONTINGENCIES

Please see Sections 12.7 and 14.4 for further details.

21.0   WASTE MANAGEMENT

This section is not applicable to this specific operating procedure.

22.0   REFERENCES

Canberra Instrumentation and Software Manuals

"*Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 2010*" DoD Environmental Quality Workgroup, Department of Navy, Lead Service.

*Further references as found in Attachment 1.

23.0   ATTACHMENTS

Attachment I

Gamma Spectroscopy Qualifications and Approvals

Basic Principles and Knowledge
Personnel shall have a basic understanding of gamma spectroscopy principles and equipment. Basic knowledge can be obtained by the review of appropriate gamma spectroscopy sections of the following references:

1. *Introduction to Health Physics* by Herman Cember

2. *Radiation Detection and Measurement* by Glen F. Knoll

3. *Measurement and Detection of Radiation* by Nicholas Tsoulfanidis

4. National Council on Radiation Protection and Measurement (NCRP) Report 58 "A Handbook of Radioactivity Measurement Procedures.

5. Vendor Courses in Gamma Spectroscopy provided by Canberra, Ortec, etc.

6. University/Academic Courses/Degrees in Health Physics, Nuclear Engineering, etc. and/or Gamma Spectroscopy

7. Other appropriate publications as approved by Cabrera Services, Inc. (List here):

Experience
Individuals operating equipment shall have sufficient experience using Canberra-based gamma spectroscopy equipment (at least one year) and/or have demonstrated successful system setup and use of such equipment under the instruction of a CABRERA Subject Matter Expert.

Approval of Qualifications
I have observed the individual perform the successful setup and use of gamma spectroscopy equipment.

Employee / Trainee:

_____
(name)

_____
(signature)

Approval:

_____
CABRERA Subject Matter Expert   Date