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Revision 0

CCP
Gamma Energy Assay (GEA) Calibration, Confirmation, and Verification Procedure

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APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	01/11/2010	Initial issue.

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LIST OF ATTACHMENTS

1.0 Purpose

This procedure contains the operating instructions to calibrate the Gamma Energy Assay (GEA) systems using the CANBERRA Genie 2000 and Non Destructive Assay (NDA 2000) Software. The GEA systems are used to quantify the activity of individual radioisotopes present in the waste sample. The assay must meet the requirements of Central Characterization Project (CCP)-PO-002, *CCP Transuranic Waste Certification Plan*, for drum shipment to the Waste Isolation Pilot Plant (WIPP).

1.1 Scope

This procedure specifies instructions to calibrate the GEA systems, which includes starting up and using the CANBERRA Genie 2000 and NDA 2000 software and calibration of the equipment.

2.0 REQUIREMENTS

2.1 References

Baseline Documents

- Canberra Industries, Inc., Publication No. 9231594F, *NDA 2000 Users Manual* (corresponding to current software version)
- Canberra Industries, Inc., Publication No. 9231595C, *NDA 2000 Technical Reference Manual* (corresponding to current software version)
- Document No. 96048, *WRAP Gamma Energy Assay System Installation and Maintenance Manual*
- ASTM C 1030, *Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectroscopy,* Annual Book of ASTM Standards, Vol. 12.01
- CCP-QP-022, *CCP Software Quality Assurance Plan*
- WRPL-OP-0907, *Gamma Energy Assay Operations Using NDA 2000*

Referenced Documents

- CCP-PO-002, *CCP Transuranic Waste Certification Plan*
- CCP-QP-002, *CCP Training and Qualification Plan*
- CCP-QP-008, *CCP Records Management*
- CCP-TP-071, *CCP Gamma Energy Assay (GEA) Operating Procedure*

2.2 Training Requirements

2.2.1 Personnel performing this procedure will be trained and qualified in accordance with CCP-QP-002, *CCP Training and Qualification Plan* prior to performing this procedure.

2.3 Precautions and Limitations

2.3.1 Physical Hazards and Precautions

[A] The germanium detectors require liquid nitrogen (LN) cooling for proper operation. Routine filling of the detector dewar ensures continuous operation of the detector. The extremely cold temperature of liquid nitrogen can cause severe burns to the skin.

[B] During normal operation, motion will start with a brief audible warning prior to any motion.

2.3.2 Radiological Hazards and Precautions

[A] Radioactive transmission sources are installed in lead and steel shields – no lead is exposed.

2.3.3 Radioactive calibration sources/standards are to be treated and controlled as sealed radioactive sources in accordance with host site procedures. Integrity tests must be performed on sources/standards per the requirements of host site procedures.

2.3.4 Emergency Shutdown Instructions

[A] Pressing the large red EMERGENCY STOP button will abort all mechanical motion, however begun assays will continue to acquire.

2.4 System Overview

NOTE

A brief description of the gamma assay based NDA systems used to determine the Transuranic (TRU) content of bulk waste and the analysis method are mentioned below, detail descriptions and analysis algorithms are listed in the NDA 2000 User's and Technical Reference Manuals.

2.4.1 The GEA systems are a high-sensitivity, shielded, gamma-ray measurement system. They have conveyor and loading systems for automatically or manually moving 55-gallon (208-liter) drums into the shield and placing them on a rotator. Four collimated standard electrode germanium (SEGe) segmented detectors view

the drum directly across from four transmission sources for determining matrix density. Measurements of individual radioisotope activities are made with and without transmission sources being exposed to the drum. Two additional low energy germanium (LEGe) isotopic detectors are positioned at right angles to the transmission sources – detector axis to determine isotopic ratios of plutonium and uranium in the waste container. Data are collected as 32k-channel spectra for segmented detectors and 4k-channel spectra for isotopic detectors.

2.4.2 The gamma assay systems shall be calibrated for the specific analytes of interest. Each calibration will employ radionuclides and waste matrices sufficient to calibrate the system for the planned application. Primary calibration standards shall be obtained from suppliers maintaining a nationally accredited measurement program. When primary standards are **NOT** available, the standards used shall be correlated with primary standards obtained from a nationally accredited measurement program. The calibration is performed once prior to assaying samples and repeated at such time that routine performance checks DO **NOT** meet acceptance criteria and the system cannot be brought into compliance.

[A] Calibration consists of the following:

[A.1] Energy and shape calibration of each germanium detector.

[A.2] Efficiency calibration of the segmented detectors with uniformly distributed activity in calibration drums of several densities.

[A.3] Transmission calibration for the segmented detectors.

2.5 Definitions

2.5.1 **Count Type** – A particular counting operation (i.e., sample counting, calibration check, or background/transmission check).

3.0 RESPONSIBILITIES

3.1 NDA Expert Analyst (EA)

3.1.1 Prepares and submits the Calibration, Confirmation, and Verification Report.

3.1.2 Prepares, issues and revises, as needed the Total Measurement Uncertainty (TMU) document based on data results.

NOTE

The NDA Operator and the NDA Lead Operator (LO) may be the same individual. The NDA LO may perform NDA Operator test and functions at any time.

3.2 NDA Lead Operator (LO)

3.2.1 Oversees equipment operation.

3.2.2 Ensures that operations are performed in accordance with CCP procedures.

3.3 NDA Operator

3.3.1 Verifies measurements are performed as required.

3.4 Facility Records Custodian

3.4.1 Receives, processes, and transmits all records generated by this procedure in accordance with CCP-QP-008, *CCP Records Management.*

4.0 PROCEDURE

NOTE

Detailed descriptions of the system hardware, as well as instrument testing, inspection and preventive maintenance requirements are discussed in the applicable Hardware Reference and Preventive Maintenance Manuals.

NOTE

Startup of the GEA systems will be performed in accordance with host site operating procedures. Where sequential order is **NOT** important, steps may be performed in a sequence other than that stated in this procedure as directed by an NDA EA. This operation assumes familiarity with CANBERRA Industries, Inc. software, including Genie 2000 and NDA 2000.

NDA EA or NDA LO

4.1 Gamma Energy and Shape Calibration

4.1.1 Verify the use of transmission sources, as applicable or observe that a set of six line sources traceable to a nationally accredited measurement program are placed into tube positions 2, 3, 4, 5, 6, and 9 or equivalent in a Q^2 style Standard Matrix 55-Gallon Drum, **AND** verify data acquisition has begun. For example, see Attachment 1, Relative Source Placement Positions in the Calibration Drums.

NOTE

The expected range of values will be documented by the NDA EA in the Calibration, Confirmation and Verification report.

4.1.2 Verify the Gain is set to approximately 0.075 kilo electron volt (keV)/channel for the GEA Isotopic detectors. This provides a range of roughly 300 keV over 4k-channels.

[A] Observe Gain, Zero, Pole Zero and other settings adjustments to optimize the Energy and Full Width Half Maximum (FWHM) calibrations.

4.1.3 Verify the Gain is set to approximately 0.1 keV/Channel for all the GEA segmented detectors. This provides a range of roughly 3.2 mega electron volt (MeV) over 32k-channels.

[A] Observe Gain, Zero, Pole Zero, and other setting adjustments to optimize the energy and FWHM calibrations.

NOTE

It is **NOT** necessary to perform a calibration of the detector sum spectrum since the NDA 2000 software automatically creates the appropriate gain-shifted calibration when it sums the detectors.

4.1.4 Observe the repeating of step 4.1.2 as necessary to ensure all the GEA Isotopic Detectors are within the following limits:

Low energy photon ÷ gain = Expected centroid ± 7 channel
High energy photon ÷ gain = Expected centroid ± 15 channels
FWHM ≤ 0.85 keV @ ≤ 129 keV
FWHM ≤ 1.25 keV @ ≥ 200 keV

4.1.5 Observe the repeating of step 4.1.3 as necessary to ensure all the GEA segmented detectors are within the following limits:

Low energy photon ÷ gain = Expected centroid ± 7 channel
High energy photon ÷ gain = Expected centroid ± 15 channels
FWHM ≤ 1.1 keV @ ≤ 129 keV
FWHM ≤ 2.20 keV @ ≥ 1173 keV

4.1.6 Verify a source certificate for the sources used is available.

4.1.7 Verify an Energy Full calibration is performed as follows:

[A] The appropriate source certificate file was used.

[B] Verify the Energy and Shape Calibration Plot for each detector is printed for inclusion in the Calibration, Confirmation, and Verification Report, or Verification Report, as applicable.

[C] Verify the Energy and FWHM report for each detector is printed for inclusion in the Calibration, Confirmation, and Verification Report, or Verification Report, as applicable.

4.2 Gamma Efficiency Calibration

NDA Operator

NOTE

Q^2 drums are homogenously loaded with matrix materials that span an approximate density range from 0.02 grams per cubic centimeter (g/cc) to 2.0 g/cc.

The efficiency calibration will require, at a minimum, a set of four calibration drums (i.e., Foam, Soft Board, Particle Board, and Sand).

NOTE

Host site will load and unload containers onto the gamma assay systems as requested by the NDA EA, NDA LO, or NDA Operator.

4.2.1 Verify a set of six line sources traceable to a nationally accredited measurement program are placed into tube positions 2, 3, 4, 5, 6, and 9 or equivalent in a Q^2 style Standard Matrix 55-Gallon Drum. For example, see Attachment 1, Relative Source Placement Positions in the Calibration Drums.

NOTE

Count times will be specified by the NDA EA to ensure that approximately 1 - 2 percent counting statistics are obtained for all major peaks (i.e., Eu-152 sources 121, 344, 788, 1112, and 1408 keV).

4.2.2 Using NDA 2000 Operations, verify the Nuclear Chemical Operators (NCO) performs an efficiency calibration count adjusting the count times as necessary until approximately 1-2 percent statistics are achieved for the major peaks (i.e., 121, 344, 788, 1112, and 1408 keV).

4.2.3 **WHEN** the system prompts for Basic Information,
THEN verify Item ID with “xxxx Eff” (i.e., Sand Eff) and Description 1 with “Efficiency Calibration w/xxxx Drum” (i.e., Efficiency Calibration w/Sand Drum) is updated.

Where: xxxx = Q^2 Matrix
Foam = Foam
Soft = Soft Board
Part = Particle Board
Sand = Sand
Conc = Concrete

4.2.4 Verify the appropriate container gross weight and density is entered, the percent full is set at 100 percent, **AND** the Certificate File associated with the calibration sources is selected.

4.2.5 Verify steps 4.2.1 through 4.2.4 for each Q^2 style Standard Matrix 55-Gallon Drum to be used for the efficiency calibration are repeated.

NOTE

The system will automatically perform a full efficiency calibration analysis at the completion of each efficiency assay.

NDA LO

4.2.6 Observe the efficiency calibration being performed using NDA 2000 Calibration Operations.

4.2.7 Verify a New Efficiency Calibration is created using the current date in the Calibration Description, in the following format:

mmddyy	(i.e., 101904)		
Where:	mm	=	Month
	dd	=	Day
	yy	=	Year

4.2.8 Verify the appropriate Efficiency Counts are selected.

4.2.9 Verify the Efficiency Curve(s) are adequate.

4.2.10 Verify the Efficiency Calibration is approved once completed.

4.2.11 Verify the New Efficiency is set as Default.

4.2.12 Verify the Efficiency Calibration Report is printed for inclusion in the Calibration, Confirmation, and Verification Report.

4.3 Transmission Source Calibration

4.3.1 Verify an appropriate container configuration, as directed by the NDA EA, is loaded correctly into the assay chamber.

4.3.2 Verify a transmission source certificate for the sources used is available.

4.3.3 Observe the transmission calibration count is performed using NDA 2000 Operations.

4.3.4 Verify the Calibration description entered when prompted, included the source(s) used.

4.3.5 Verify the Transmission Calibration is approved.

4.3.6 Verify the Transmission Calibration is set as Default.

4.3.7 Verify the Transmission Calibration Report is printed for inclusion in the Calibration, Confirmation, and Verification Report.

4.4 Calibration Verification

NOTE

Calibration Verification SHALL be performed using at least one source and matrix configuration. Calibration source standards or secondary source standards that have been correlated with the calibration source standards may be used. The Daily Performance Drum Check drum, NDA-QC1, will be used for verification measurements unless otherwise directed by the NDA EA. Certain activity-based verification measurements, such as detector efficiency, may require the use of gamma line sources.

Calibration Verification SHALL be performed after any one of the following conditions has occurred:

- Major system repairs and/or modifications
- Replacement of measurement major system's components, (e.g., detector, neutron generator or supporting electronic components) that have the capacity to affect data
- Significant changes to the system's software
- Relocation of the system

The NDA EA will provide the detailed information concerning the number and strength of the sources, the matrix drum configurations, and the number of replicates.

NDA Operator

4.4.1 Verify the appropriate source and matrix drum configuration, as directed by the NDA EA, is loaded correctly into the assay chamber.

4.4.2 Verify the appropriate nuclide library is selected for the intended count type used for the verification measurements, as necessary.

4.4.3 Verify the replicate measurement(s) for each source/matrix configuration is performed as follows:

[A] Verify the appropriate assay count type (i.e., Debris or Debris Shielded) using NDA 2000 is selected.

[B] Verify that the correct certification/declaration is selected.

[C] Verify each assay report is printed for inclusion in the Calibration, Confirmation, and Verification Report or Verification Report, as applicable.

4.4.4 Verify the appropriate nuclide library is restored for the count type used, as necessary.

NDA EA

4.4.5 Evaluate the verification measurement results to determine the statistical agreement (Z-Test ≤ 1.96) with the source activity or daily performance check acceptance criteria identified in the systems operating procedure, as applicable.

4.4.6 **IF** the criteria are met, **AND** the calibration verified,
THEN document the results in a Verification Report.

4.4.7 **IF** the criteria are **NOT** met,
THEN evaluate the cause, correct, **AND** retest as needed.

4.5 Calibration Confirmation

NOTE

Calibration Confirmation SHALL be performed using nationally recognized standards, or certified standards derived from nationally recognized standards that span the range of use. The standards used to calculate accuracy SHALL **NOT** be the same as those used for the system calibration.

In order to confirm that the calibration of the NDA system was correctly established, the accuracy and precision of the system are determined after each calibration or re-calibration by performing replicate measurements of a non-interfering matrix. Calibration confirmation replicate measurements SHALL be performed on containers of the same nominal size as those in which actual waste is assayed and according to approved waste assay procedures. The number of replicate measurements to be performed SHALL be documented and technically justified. Accuracy is reported as percent recovery (%R). The applicable range for accuracy SHALL **NOT** exceed ± 30 percent on a non-interfering matrix. Precision is reported as percent relative standard deviation (%RSD). The %RSD SHALL **NOT** exceed the values listed in Table A-3.2, Upper Limits for %RSD vs. Number of Replicates of CCP-PO-002, the corresponding number of replicate measurements in a non-interfering matrix.

The NDA EA will provide the detailed information concerning the number and strength of the sources, the matrix drum configurations and the number of replicates.

NDA Operator

4.5.1 Verify the appropriate source and matrix drum configuration, as directed by the NDA EA, is loaded correctly into the assay chamber.

4.5.2 Verify replicate measurement(s) for each source matrix configuration are performed as follows:

[A] Verify the appropriate assay count type (i.e., Debris or Debris Shielded) using NDA 2000 is selected.

[B] Verify that the correct certification/declaration is selected.

[C] Verify each assay report is printed for inclusion in the Calibration, Confirmation, and Verification Report.

NDA EA

4.5.3 Evaluate the results using the calibration confirmation criteria specified in CCP-PO-002.

4.5.4 **IF** the criteria are met **AND** the calibration is confirmed,
THEN document the results in a Calibration, Confirmation, and Verification Report.

4.5.5 **IF** the criteria are **NOT** met,
THEN document the results in a Calibration, Confirmation, and Verification Report.

4.5.6 Create, edit or revise as necessary TMU documentation based on data results.

4.6 Lower Limit of Detection Determination

NOTE

The lower limit of detection (LLD) for each radioassay system must be determined. Instruments performing TRU/low-level waste discrimination measurements must have an LLD of 100 nanocuries per gram (nCi/g) or less. Site specific environmental background and container specific interferences must be factored into LLD determinations. The LLD is that level of radioactivity which, if present, yields a measured value greater than the critical level with a 95 percent probability, where the critical level is defined as that value which measurements of the background will exceed with five percent probability. Because the LLD is a measurement-based parameter, it is not feasible to calculate LLDs for radionuclides that are not determined primarily by measurement, (e.g., Sr-90). In such cases, the site shall derive the equivalent of an LLD, i.e., a reporting threshold for a radionuclide(s), when it is technically justified. This value may be based on decay kinetics, scaling factors, or other scientifically based relationships and must be adequately documented in site records. For purposes of reporting radionuclide data in the Waste Isolation Pilot Plant Waste Information System and Waste Data System (WWIS/WDS), this value will be the equivalent of an LLD.

NOTE

The NDA EA will provide information concerning the matrix drum configuration(s) to be used (i.e., Foam, Soft Board, Particle Board, or Sand or concrete drums, as applicable), and the number of replicates required.

NDA Operator

4.6.1 Verify the appropriate matrix drum configuration, as directed by the NDA EA, is loaded correctly into the assay chamber.

4.6.2 Verify the replicate measurement(s) for each matrix configuration is performed as follows:

[A] Verify the appropriate assay count type (i.e., Debris or Debris Shielded) using NDA 2000 is selected.

[B] Verify that the correct certification/declaration is selected.

[C] Verify each assay report is printed for inclusion in the Calibration, Confirmation and Verification Report, as applicable.

4.7 Measurement Control

NOTE

To demonstrate that data of known and documented quality are generated by measurement systems, a series of quality control (QC) measurements may be established and implemented. These QC measurements include daily environmental and QC background measurements, daily performance checks, and weekly interfering matrix checks, as applicable.

A series of measurements is required to establish QC limits and/or boundaries for the applicable daily environmental and QC background measurements, daily performance checks, and weekly interfering matrix checks.

The NDA EA will provide the detailed information concerning the number and strength of the sources, the matrix drum configurations and the number of replicates required.

4.7.1 Verify the appropriate source and matrix drum configuration, as directed by the NDA EA, is loaded correctly into the assay chamber.

4.7.2 Verify the replicate measurements for each source and/or matrix configuration are performed as follows:

[A] Verify the appropriate assay count type (i.e., Debris or Debris Shielded) using NDA 2000 is selected.

[B] Verify that the correct certification/declaration is selected.

[C] Verify each assay report is printed for inclusion in the Calibration, Confirmation, and Verification Report or Verification Report, as applicable.

4.8 Diagnostics Measurements

4.8.1 Additional measurements may be performed for data gathering and/or diagnostics purposes using the previously described sources, containers and configurations under the direction of the NDA EA as necessary.

4.9 Records Transmittal

4.9.1 Transfer all records generated by this procedure to the Facility Records Custodian.

Facility Records Custodian

4.9.2 Receive, process, and transmit all records generated by this procedure in accordance with CCP-QP-008.

5.0 RECORDS

5.1 Records generated during the performance of this procedure are maintained as QA records in accordance with CCP-QP-008. The records are the following:

5.1.1 QA/Nonpermanent

[A] Calibration, Confirmation, and Verification Report

[B] TMU Document

[C] Verification Report

Attachment 1 – Relative Source Placement Positions in the Calibration Drums (EXAMPLE)