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CCP-TP-010

Revision 3

CCP
Waste Assay Gamma Spectrometer (WAGS) and SWEPP Gamma-Ray Spectrometer (SGRS) Calibration Procedure

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PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	02/26/2005	Initial issue.
1	04/15/2005	Addressed CBFO adequacy review comments.
2	06/15/2006	Revised to address corrective actions identified in response to Carlsbad Field Office (CBFO) Corrective Action Report (CAR) CAR-06-022.
3	11/06/2006	Revised to enhance reference peak and energy calibrations and to extend efficiency calibration range to include higher density material.

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LIST OF ATTACHMENTS

1.0 PURPOSE

This procedure contains the operating instructions to calibrate the Waste Assay Gamma Spectrometer (WAGS) and Stored Waste Examination Pilot Plant (SWEPP) Gamma-Ray Spectrometer (SGRS) Systems using the Canberra Genie 2000 and Non Destructive Assay (NDA2000) Software. The WAGS and SGRS systems are used to quantify the activity of individual radioisotopes present in the waste sample. The assay must meet the requirements of 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 WAGS and SGRS systems, which includes starting up and using the Canberra Genie 2000 and NDA2000 software and calibration of the equipment.

2.0 REQUIREMENTS

2.1 References

Baseline Documents

- Document No. 99092, Rev. C, Automatic Q2 System with 6 Drum Conveyor*, Hardware Reference Manual*
- ASTM C 1030, *Standard 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*

Referenced Documents

- Canberra Industries, Inc., Publication No. 9231594F, *NDA2000 Users Manual* (corresponding to current software version)
- Canberra Industries, Inc., Publication No. 9231595C, *NDA2000 Technical Reference Manual* (corresponding to current software version)
- CCP-PO-002, *CCP Transuranic Waste Certification Plan*
- CCP-QP-002, *CCP Training and Qualification Plan*
- CCP-QP-008, *CCP Records Management*

- CCP-TP-019, *CCP Waste Assay Gamma Spectrometer* (WAGS) *Operating Procedure*
- CCP-TP-115, *CCP SWEPP Gamma-Ray Spectrometer (SGRS) Operating Procedure*
- MP-RS&C-6.16, *Radioactive (Non-Nuclear) Source Control*

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 Equipment List

2.3.1 Broad Energy Germanium (BEGe) Detectors

2.3.2 Conveyor mechanism (WAGS)

2.3.3 Sample rotator

2.3.4 Transmission sources (WAGS)

2.3.5 Shutter assembly (WAGS)

2.3.6 An electronics rack housing:

[A] Reference (quad) pulsers or equivalent

[B] Digital Spectrum Analyzer (DSA1000) units or equivalent

[C] An operator's terminal including:

[C.1] Personal computer with appropriate software

[C.2] Computer keyboard

[C.3] Computer mouse

[C.4] Computer monitor

[C.5] Printer

[D] Uninterruptible power supplies (UPSs)

2.3.7 Software

[A] NDA2000, Waste Assay

[B] Genie 2000, Gamma Acquisition and Analysis

2.4 Precautions and Limitations

2.4.1 Physical Hazards and Precautions

[A] The BEGe 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 of the WAGS will start with an audible warning five seconds prior to any motion.

2.4.2 Radiological Hazards and Precautions

[A] The WAGS has three radioactive transmission sources installed in a lead and steel shield - no lead is exposed.

2.4.3 Radioactive calibration sources/standards are to be treated and controlled as sealed radioactive sources in accordance with MP-RS&C-6.16, *Radioactive (Non-Nuclear) Source Control*. Integrity tests must be performed on sources/standards per the requirements of MP-RS&C-6.16.

2.4.4 Emergency Shutdown Instructions

[A] Pressing the large red EMERGENCY STOP button on the Programmable Logic Controller (PLC), Conveyor, or Assay Chamber will abort the WAGS operation.

[B] Pressing the large red EMERGENCY STOP button on the PLC mounted on the side of the SGRS assay chamber will STOP operation of the SGRS rotator.

2.5 System Overview

2.5.1 The WAGS system is a gamma spectroscopy based NDA system to determine the transuranic (TRU) content of bulk waste. A brief description of the NDA system and the analysis method follows. (Detailed descriptions and analysis algorithms are listed in the NDA2000 User's and Technical Reference Manuals.)

[A] The WAGS system is a high-sensitivity, shielded, Gamma-Ray measurement system. It has a conveyor and loading system for automatically or manually moving 55-gallon (208 liter) drums into the shield and placing them on a rotator. Three uncollimated BEGe segmented detectors view the drum directly across from three transmission sources for determining matrix density. Measurements of individual radioisotope activities are made with and without transmission sources being exposed to the drum. Three additional BEGe 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 16k-channel spectra for each detector.

2.5.2 The SGRS system is a gamma spectroscopy based NDA system to determine the TRU content of bulk waste. A brief description of the NDA system and the analysis method follows. (Detailed descriptions and analysis algorithms are listed in the NDA2000 User's and Technical Reference Manuals.)

[A] The SGRS is a high-sensitivity, shielded Gamma-Ray measurement system, which employs only the summed spectrum multi-curve density measurement methodology. Four detectors are used on the summed spectrum analysis and to measure the plutonium isotopic distribution of waste items. Data are collected as 16k-channel spectra for each detector.

2.5.3 The WAGS and SGRS 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 BEGe detector.

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

[A.3] Transmission calibration with an empty drum for the segmented detectors (WAGS).

[A.4] Reference peak calibration with a highly-stable pulser for live time correction.

2.6 Definitions

2.6.1 **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 and issues the Total Measurement Uncertainty (TMU) document.

NOTE

The NDA Operator and the NDA Lead Operator (LO) may be the same individual. The 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 operations are performed in accordance with CCP procedures.

3.3 NDA Operator

3.3.1 Conducts measurements 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 WAGS or SGRS will be performed in accordance with CCP-TP-019, *CCP Waste Assay Gamma Spectrometer* (WAGS) *Operating Procedure* and CCP-TP-115, *CCP SWEPP Gamma-Ray Spectrometer* (SGRS) *Operating Procedure*. Where sequential order is **NOT** important, steps may be performed in a sequence other than that stated in this procedure as directed by an EA. This operation assumes familiarity with Canberra Industries, Inc. software, including Genie 2000 and NDA2000.

NOTE

This procedure contains instructions to calibrate the WAGS and SGRS Systems. Only the steps applicable to the system being calibrated need to be performed.

NDA EA or LO

4.1 Gamma Energy and Shape Calibration

4.1.1 Place a set of six Eu-152/Am-241 line sources traceable to a nationally accredited measurement program into tube positions 2, 3, 4, 5, 6, and 9 or equivalent in a Q^2 style Standard Matrix 55-Gallon Drum, and begin acquiring data. For example, see Attachment 1, Relative Source Placement Positions in the Calibration Drums.

4.1.2 Set the gain to approximately 0.125 kilo electron volts (keV)/channel for the WAGS Segmented Detectors, which provides a range of roughly 2.0 mega electron volts (MeV) over 16k-channels.

NOTE
The expected range of values will be document by the EA.

[A] As applicable, adjust the Gain, Zero, Pole Zero and other settings to optimize the Energy and Full Width at Half Maximum (FWHM) calibrations.

[A.1] **IF** Gain, Zero, Pole Zero, **OR** other settings are adjusted,
THEN ensure the reference peak calibration is within the expected range.

4.1.3 Set the Gain to approximately 0.075 keV/channel for the WAGS Isotopic Detectors. This provides a range of roughly 1.2MeV over 16k-channels.

[A] As applicable, adjust the Gain, Zero, Pole Zero and other settings to optimize the Energy and FWHM calibrations.

4.1.4 Set the Gain to approximately 0.090 keV/Channel for all the SGRS Detectors. This provides a range of roughly 1.5MeV or 16k-channels.

[A] As applicable, adjust Gain, Zero, Pole Zero, and other settings to optimize the energy and FWHM calibrations.

[A.1] **IF** Gain, Zero, Pole Zero, **OR** other settings are adjusted,
THEN ensure the reference peak calibration is within the expected range.

NOTE

It is **NOT** necessary to perform a calibration of the detector sum spectrum since the NDA2000 software automatically creates the appropriate gain-shifted calibration when it sums the detectors.

4.1.5 Repeat step 4.1.2 as necessary to ensure all the WAGS Segmented detectors are within the following limits:

121.8 keV	=	974 ± 1 channel
1408.0 keV	=	11264 ± 2 channels
FWHM	≤	0.85 keV @ 121.8 keV
FWHM	≤	2.20keV @ 1408.0 keV

4.1.6 Repeat step 4.1.3 as necessary to ensure all the WAGS Isotopic Detectors are within the following limits:

121.8keV	=	1624 ± 1 channel
1112.1keV	=	14827 ± 2 channels
FWHM	≤	0.85 keV @ 121.8keV
FWHM	≤	2.20 keV @ 1112.1keV

4.1.7 Repeat step 4.1.4 as necessary to ensure all the SGRS Detectors are within the following limits:

121.8 keV	=	1353 ± 1 channel
1408.0 keV	=	15644 ± 2 channels
FWHM	≤	0.85 keV @ 121.8 keV
FWHM	≤	2.20 keV @ 1408.0 keV

4.1.8 Create a source certificate for the Eu-152/Am-241 line sources installed, as required.

4.1.9 Using Genie 2000 perform an Energy Full calibration:

[A] Select the certificate file associated with the line sources used.

[B] Print out each Energy and Shape Calibration Plot for inclusion in the Calibration, Confirmation, and Verification Report, or Verification Report, as applicable.

[C] Print out each Energy and FWHM report 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 a 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 as requested by the NDA EA, LO, or Operator.

4.2.1 Place a set of six Eu-152/Am-241 line sources traceable to a nationally accredited measurement program 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 for Source Placement.

NOTE

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

4.2.2 Using NDA2000 Operations, perform an efficiency calibration count adjusting the count times as necessary until approximately 1% 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 update 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).

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

4.2.4 Enter the gross weight, density, ensure percent full is set at 100%, **AND** select the Certificate File associated with the calibration sources.

4.2.5 Repeat 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.

NOTE

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

NDA LO

4.2.6 Perform an efficiency calibration using NDA2000 Calibration Operations.

4.2.7 Create a New Efficiency Calibration 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 Select the appropriate Efficiency Counts.

4.2.9 Select the appropriate Efficiency Curve(s).

4.2.10 Approve the Efficiency Calibration once completed.

4.2.11 Set the new Efficiency as Default.

4.2.12 Print out the Efficiency Calibration Report for inclusion in the Calibration, Confirmation, and Verification Report.

4.3 Transmission Source Calibration (WAGS)

4.3.1 Place an empty 55-gallon drum into the assay chamber.

4.3.2 Create a transmission source certificate, as required.

4.3.3 Perform a transmission calibration count using NDA2000 Operations.

4.3.4 Enter the Calibration description when prompted, including the source(s) used.

4.3.5 Approve the Transmission Calibration.

4.3.6 Set the Transmission Calibration as Default.

4.3.7 Print out the Transmission Calibration Report for inclusion in the Calibration, Confirmation, and Verification Report.

4.4 Reference Peak Calibration

NOTE
This procedure must be performed for each detector that has a reference pulser. The reference pulser is used to correct data for live time losses.

4.4.1 Setup each applicable reference peak output as follows:

[A] Set pulser output frequency to 100Hz.

[B] Set pulser output amplitude to produce a peak, in a spectral region where there are no Gamma-Ray peak interferences as assigned by the EA.

4.4.2 Using Genie 2000 Gamma Acquisition and Analysis, acquire a spectrum of the reference peak, **AND** adjust the count times as necessary until approximately 1% statistics are reached.

[A] Perform analysis for the peak location.

[A.1] Select ANALYZE → Peak Locate → Unidentified 2nd Diff….

[A.2] Select the GENERATE REPORT check box.

[A.3] Select EXECUTE.

[B] Perform analysis for the peak area.

[B.1] Select ANALYZE → Peak Area → Sum / Non-Linear LSQ fit…

[B.2] Select the GENERATE REPORT check box.

[B.3] Select EXECUTE.

[C] Review report, **AND** print report window for inclusion in the Calibration, Confirmation and Verification Report or Verification Report, as applicable.

4.4.3 Repeat step 4.4.2 for each applicable detector.

4.4.4 Using NDA2000 Reference Peak, perform a Reference Peak Calibration.

[A] OPEN Datasource (i.e. Select File → Open), change source from CAM File to Detector, Select, **AND** OPEN an appropriate detector.

[B] Ensure the Select as REFERENCE checkbox is **NOT** set.

[C] Select the appropriate PEAK in the Peak Analysis Results table.

[D] Set the Select as REFERENCE checkbox and ensure the Energy, Rate and Error values are updated with the values from the selected Peak Analysis Results peak.

[E] Set Source Type to PULSER and SAVE the file using the disk icon on the toolbar.

4.4.5 Repeat step 4.4.4 for each applicable detector.

4.5 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, DPC 9999, will be used for verification measurements unless otherwise directed by the 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 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 Obtain the appropriate source and matrix drum configuration as directed by the EA.

4.5.2 Position the source(s) within the matrix as applicable.

4.5.3 Perform replicates for each source/matrix configuration as follows:

[A] Select appropriate assay count type (i.e., Assay-Passive, Assay-Debris or Assay-Solid/Soils) using NDA2000.

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

[C] Print out the assay report for inclusion in the Calibration, Confirmation and Verification Report or Verification Report, as applicable.

NDA EA

4.5.4 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.

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

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

4.6 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% 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 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

4.6.1 Obtain the appropriate source and matrix drum configuration.

4.6.2 Position the source(s) within the non-interfering matrix drum.

4.6.3 Perform replicate measurements for each source matrix configuration as follows:

[A] Select appropriate assay count type (i.e., Assay-Passive, Assay-Debris or Assay-Solid/Soils) using NDA2000.

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

[C] Print out the assay report for inclusion in the Calibration, Confirmation and Verification Report.

NDA EA

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

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

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

4.7 Records Transmittal

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

Facility Records Custodian

4.7.2 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 quality assurance (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)