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CCP-TP-167

Revision 1

CCP
Drum Waste Assay System Imaging Passive/Active Neutron Calibration

EFFECTIVE DATE: 06/19/2008

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PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	10/12/2007	Initial issue.
1	06/19/2008	Revised to incorporate editorial auditor comments from U.S. Department of Energy (DOE), Carlsbad Field Office (CBFO) Certification Audit A-08-06 and Environmental Protection Agency (EPA) Inspection Number EPA-ORNL-CCP-CH-11.07-8 of November 2007.

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LIST OF ATTACHMENTS

1.0 PURPOSE

This procedure describes the process for calibrating the Drum Waste Assay System (DWAS) Imaging Passive/Active Neutron (IPAN) system. The range of applicability is specified in CCP-TP-166, *CCP Drum Waste Assay System Imaging Passive/Active Neutron Operations*.

1.1 Scope

The procedure is applicable to all personnel responsible for operations of the DWAS IPAN or analysis of the DWAS IPAN data.

2.0 REQUIREMENTS

2.1 References

Baseline Documents

- NUREG/CR-5550, LA-UR-90-732, *Passive Non-Destructive Assay of Nuclear Materials*

Referenced Documents

- ASTM Test Method C1493-01, *Standard Test Method for Non-Destructive Assay of Nuclear Material in Waste by Passive and Active Neutron Counting Using a Differential Die-Away System*.
- ASTM Method E181-98, *Standard Test Methods for Detector Calibration and Analysis of Radionuclides*.
- BII-5183-CVR-001, *DWAS IPAN Calibration and Validation Report*
- BII-TMU-5183-001, *DWAS IPAN Total Measurement Uncertainty Report*
- CCP-QP-002, *CCP Training and Qualification Plan*
- CCP-QP-008, *CCP Records Management*
- CCP-QP-022*, CCP Software Quality Assurance Plan*
- CCP-TP-166, *CCP Drum Waste Assay System Imaging Passive/Active Neutron Operations*

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 IPAN Assay

- Turntable
- Neutron Generator System
- ^{3}He Neutron Detectors

2.3.2 Data Acquisition System Electronics Rack (see Attachment 1, Hardware and Software Settings, in CCP-TP-166 for an example)

2.3.3 Control Cabinet

2.3.4 Analysis Computer and ancillary equipment, such as monitors and printers

2.3.5 Interfering and non-interfering surrogate matrix drums

2.4 Software

2.4.1 DWAS.EXE

2.4.2 DWASAnalysis.EXE

2.4.3 DNGI.EXE (DWAS Neutron Gamma Integration)

2.4.4 DWAS_SGS_QC.XLS

2.4.5 FRAM44.EXE (Fixed energy, Response function Analysis with Multiple efficiencies)

2.5 Precautions and Limitations

2.5.1 Radiological Hazards and Precautions

[A] The assay room will **NOT** be entered during neutron generator operation. The neutron generator will **NOT** be approached during neutron generator operation. The system is capable of producing $1x10^6$ neutrons at 14 million electron volts (MeV) per pulse. The system is designed to be operated from the control room only and as low as reasonably achievable (ALARA) dose practices should be provided for all personnel in other nearby areas as determined by qualified Host site Health Physics personnel.

[B] During normal operation, motion of the system will start without audible warning. Access to the assay room shall be controlled by the Nondestructive Assay (NDA) Operator during normal operation and when performing maintenance and repairs.

[C] The instrument will be located in an area with a low ambient radiation background. The movement of radioactive materials in the vicinity of the system will be minimized while a measurement is in progress.

2.6 Definitions

2.6.1 **Daily** – Once each day the DWAS IPAN is used.

2.6.2 **Operational Week** – Any week (7 consecutive days) the DWAS IPAN is used.

3.0 RESPONSIBILITIES

3.1 Nondestructive Assay (NDA) Operator

3.1.1 Collects and records data on Attachment 1, Calibration Log Sheet, for passive/active neutron calibration or recalibration, confirmation of calibration, and verification of calibration, as required.

3.1.2 Ensure all software is approved in accordance with CCP-QP-022, *CCP Software Quality Assurance Plan,* and on the Software Inventory List (SIL) before use.

3.2 NDA Lead Operator (LO)

3.2.1 Provides supervision of the overall operation of the NDA system.

3.2.2 Ensures that this procedure is implemented and followed by operations and analysis personnel.

3.2.3 Reviews Attachment 1 and associated data.

3.3 NDA Expert Analyst (EA)

3.3.1 Provides technical support, as needed, and is responsible for reviewing data produced by this procedure.

3.3.2 Performs Passive/Active Neutron validation.

3.3.3 Prepares the Calibration and Validation Report.

3.4 Facility Records Custodian

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

4.0 PROCEDURE

NOTE

Calibration of the DWAS IPAN is performed and documented as described in this procedure. This is in accordance with the principles, as they apply, set forth in American Society for Testing and Materials (ASTM) Test Method C1493-01, *Standard Test Method for Non-Destructive Assay of Nuclear Material in Waste by Passive and Active Neutron Counting Using a Differential Die-Away System* and ASTM Test Method E181-98, *Standard Test Methods for Detector Calibration and Analysis of Radionuclides*. Each calibration will employ radionuclides and waste matrices sufficient to calibrate the system for the planned application. Calibration standards SHALL be traceable to suppliers maintaining an accredited measurement program. NDA Performance Demonstration Program (PDP) standards may **NOT** be used as primary calibration standards but may be used for confirmation measurements.

NOTE

Passive and active neutron calibration, validation, confirmation, and verification results will be summarized and documented on Attachment 1, as required.

NOTE

A passive/active neutron calibration is performed once prior to assaying production drums and SHALL be performed when any of the following conditions exist:

- A change to system configuration has been made that affects the system response.
- The intended application is outside the current range of the calibration.
- Performance checks in accordance with CCP-TP-166 indicate the system is out of tolerance after two successive runs, troubleshooting activities fail to correct the problem, **AND** the NDA EA or NDA LO recommends recalibration.

4.1 Passive/Active Neutron Calibration or Recalibration

NDA Operator

NOTE

The following information will be recorded in the DWAS.EXE software per CCP-TP-166 at the time calibration measurements are performed:

- Primary ID: Source ID, Radius, Height
- Secondary ID: Matrix ID (e.g. NDA-5183-001)

4.1.1 Initiate an Attachment 1 by circling Calibration at the top, **AND** record the Facility on Attachment 1.

NOTE

Sources are chosen so that they produce sufficient counting statistics when placed in the calibration matrix. For active neutron calibration Depleted U sources are used as surrogates for Pu-239. For passive neutron calibration, Cf-252 sources are used as surrogates for Pu-240. Passive neutron and active neutron calibration must be completed separately. The Depleted U **AND** Cf-252 sources must **NOT** be placed in the matrix at the same time.

4.1.2 Obtain the sources specified by the NDA EA or NDA LO.

4.1.3 Document on Attachment 1 the source identification.

NOTE

For source location, radius is the position in inches of the source from the center of the matrix drum and height is the position, in inches, from the bottom of the source to the bottom of the matrix drum.

NOTE

The number of positions should **NOT** exceed the number of detector groups.

NOTE

Source positions are chosen based on the resolution required to obtain the performance desired. Nine (9) positions are typical for the DWAS IPAN.

4.1.4 Obtain a list of Source Positions and System Settings required for calibration from the NDA EA or NDA LO.

4.1.5 Document the Source Positions on Attachment 1 in the Radius and Height columns for each position number.

NOTE

Matrix mockups will be chosen to simulate the neutron properties of actual waste that will be assayed by the instrument. The NDA LO will provide material parameters (i.e., weight, matrix type, etc.) for each matrix drum.

4.1.6 Obtain the matrix specified by the NDA EA or NDA LO.

4.1.7 Document matrix identification on Attachment 1.

4.1.8 Obtain **AND** document the Gross, Tare, and Net Weights (in kg) on Attachment 1.

NOTE

Final Reports generated for Instrument Performance Background and Source Measurements in this procedure will be included in the Calibration and Validation Report.

4.1.9 Perform Instrument Performance Background and Source Measurements in accordance with CCP-TP-166.

NOTE

A No Source Background matrix drum measurement is required at least once each shift for the calibration matrix of interest. For example, if a Dry Combustibles matrix is being used for calibration, the matrix should be assayed without a source in place. These runs are commonly labeled NSBKG for No Source Background.

4.1.10 Complete all operational requirements in accordance with CCP-TP-166, **AND** indicate on Attachment 1:

[A] No Source Background matrix drum measurements. (NSBKG)

[B] Source position matrix drum measurements (Position #).

4.1.11 Contact the NDA EA or NDA LO for more information or clarification on any of the above items.

4.1.12 Review Attachment 1, **AND** print name, sign, and date Attachment 1.

4.1.13 Provide electronic Calibration Data Files, **AND** Attachment 1(s) to the NDA LO for review.

NDA LO

4.1.14 Review Attachment 1, **AND** print name, sign, and date.

4.1.15 Transmit electronic Calibration Data Files, **AND** reviewed Attachment 1(s) to the NDA EA.

NDA EA

4.1.16 Document the calibration results in the Calibration and Validation Report.

4.2 Passive/Active Neutron Calibration Library Update

NDA EA

4.2.1 Obtain the data taken during calibration activities.

NOTE

During calibration, preliminary measurement control limits will be based on either initial factory measurements or measurements performed during the beginning of the calibration process. The EA will determine the acceptability of the measurements when compared to these values. These measurements and results will be documented in the NDA Operational Logbook.

4.2.2 Verify that instrument performance source and background measurements taken during calibration fall within set or preliminary parameters.

4.2.3 Review individual measurements for acceptability when compared to the preliminary measurement control data.

NOTE

Once the data have been confirmed as valid and formatted for entry into the calibration libraries, new libraries are generated in accordance with CCP-QP-022.

4.2.4 Prepare the data in the appropriate format for .amx and .pmx files.

4.2.5 Update the .amx and .pmx software library files in accordance with CCP-QP-022.

4.3 Passive/Active Neutron Validation

NDA EA

NOTE

Calibration validation should be completed following the completion of calibration measurements. Validation is used as a first order quality assurance (QA) check of the new calibration library to confirm that the calibration data used for the library was acceptable. First order QA verifies that the results of the processed calibration data do not contain any errors. The procedure for a first order QA check replays the calibration data through the implemented algorithms and uses the Pu-239 effective mass (active) and Pu-240 equivalent mass (passive) to compare with the respective effective mass of the radioactive standard used for calibration. Because the data being analyzed are the same data used in establishing the calibration constants, the actual effective mass and measured effective mass will be the same.

4.3.1 Initiate a new Attachment 1 by circling Validation at the top, **AND** record the Facility on Attachment 1.

4.3.2 Obtain the original calibration data and calibration libraries to be tested as specified by the NDA EA or NDA LO, **AND** copy onto Attachment 1.

4.3.3 Select a measurement on Attachment 1, **AND** calculate final radioassay results, using DNGI.exe.

4.3.4 Verify the measured Pu-240 equivalent mass and/or the Pu-239 effective mass is the same (+\- 5%) as the radioisotopic source used for calibration.

4.3.5 **IF** the values are the same (+\-5%),
THEN indicate Pass in the associated QA Check column on Attachment 1,
ELSE indicate Fail.

4.3.6 Repeat steps 4.3.3 through 4.3.5 until all calibration measurements have been validated.

NOTE

If all results have passed, then the calibration has been validated and is ready for use.

4.3.7 Print name, sign, and date Attachment 1, **AND** forward to NDA LO for review.

NDA LO

NOTE

If all validation runs fall within the required range, then the new calibration libraries are accurate, and can be released in accordance with CCP-QP-022 for use with production assays.

4.3.8 Review Attachment 1, **AND** print name, sign, and date.

4.3.9 Transmit completed Attachment 1 to the Facility Records Custodian.

NDA EA

4.3.10 Document the validation results in the Calibration and Validation Report.

4.4 Confirmation of Calibration

NDA Operator

NOTE

Calibration Confirmation SHALL be performed after each Passive/Active Neutron Calibration or recalibration using at least one non-interfering matrix. Matrices representative of the calibration range may also be tested as required.

A minimum of two source strengths or loadings are required: One to cover the lower one-third of the operating range and one to cover the upper one-third of the operating range. Sources used for calibration will **NOT** be used for confirmation.

4.4.1 Obtain the appropriate source/matrix configuration as indicated by NDA LO.

4.4.2 Initiate a new Attachment 1 by circling Confirmation at the top, **AND** recording the Facility on Attachment 1.

4.4.3 Verify that the source standard(s) is traceable to suppliers maintaining an accredited measurement program.

4.4.4 Position the source(s) within the matrix as indicated by the NDA LO.

4.4.5 Perform background and source instrument performance checks as described in CCP-TP-166.

NOTE

The number of replicates to be run will be decided by the NDA EA based on the data contained in the Calibration and Validation Report.

4.4.6 Perform replicates for each source/matrix configuration as indicated by the NDA LO per the standard assay procedure described in CCP-TP-166.

4.4.7 Verify all required information is documented on Attachment 1.

4.4.8 Compare the mean measured value for the gram equivalents of Pu-240 or the Pu-239 effective mass, as appropriate, for the replicate measurements to the tag values of the respective measures for the source(s), **AND** indicate on Attachment 2, Calibration Verification Sheet.

4.4.9 Print name, sign, and date Attachments 1 and 2.

4.4.10 Provide data and Attachments 1 and 2 to the NDA LO for review.

NOTE

Signature by reviewer indicates all calculations have been validated.

NDA LO

4.4.11 Review Attachments 1 and 2, **AND** print name, sign, and date.

4.4.12 Provide data and Attachments 1 and 2 to the NDA EA.

NDA EA

4.4.13 Document the confirmation results in the Calibration and Validation Report.

4.4.14 Transmit Calibration and Validation Report to the Facility Records Custodian.

NOTE

For Calibration Confirmation, accuracy SHALL **NOT** exceed +/-30 percent. The required precision is reported as percent relative standard deviation (%RSD) **AND** SHALL **NOT** exceed the values listed in Table 1 below.

Table 1. Upper Limits for %RSD vs. Number of Replicates[a]

Number of Replicates	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Max %RSD	1.8	6.6	10.0	12.3	14.0	15.2	16.2	17.1	17.7	18.3	18.8	19.3	19.7	20.0

[a] The values listed are derived from the measured standard deviation of the replicate measurements using $\frac{s}{\mu} \bullet 100\% < \sqrt{\frac{(0.292) \bullet \chi^2_{0.05, n-1}}{n-1}} \bullet 100\%$ where s is the measured standard deviation, n is the number of replicates, μ is the true value. $\chi^2_{0.05, n-1}$ is the critical value for the upper 5 percent tail of a one sided chi-squared distribution with n-1 degrees of freedom, and 0.292 corresponds to a 95 percent upper confidence bound on the true system precision limit of 29.2 percent.

4.4.15 Transmit completed Attachments 1 and 2 to Facility Records Custodian.

NDA Operator

4.5 Verification of Calibration

NOTE

Verification of the passive and active calibration is performed using at least one source/non-interfering matrix configuration. Calibration source standards or secondary source standards that have been correlated with the calibration source standards can be used. Sources used SHALL **NOT** be the same sources used for calibration.

Calibration Verification should be performed after any one of the following occurs:

- Failure of the Daily Instrument Performance Source Measurement as described in CCP-TP-166
- Major system repairs and/or modifications
- Replacement of the measurement 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

4.5.1 Obtain the appropriate source/non-interfering matrix (e.g., daily calibration verification source and empty drum) configuration as indicated by NDA LO.

4.5.2 Initiate a new Attachment 1 by circling Verification at the top, **AND** record the Facility on Attachment 1.

4.5.3 Verify that the source standard(s) is traceable to suppliers maintaining an accredited measurement program or is a secondary source standard(s) that has been correlated with the calibration source standard(s).

4.5.4 Position the source(s) within the matrix as indicated by the NDA LO.

[A] Contact the NDA LO for advice on the best way to secure the source in place.

4.5.5 Verify the daily Instrument Performance Background Measurement has been completed as described in CCP-TP-166.

4.5.6 Verify the daily Instrument Performance Source Measurement has been completed as described in CCP-TP-166.

4.5.7 **IF** either steps 4.5.5 or 4.5.6 can **NOT** be verified, **THEN** complete the measurement as described in CCP-TP-166.

NOTE

The number of replicates to be run will be decided by the NDA EA based on the data contained in the Calibration and Validation Report.

4.5.8 Perform replicates for each source/matrix configuration using the counting time indicated by the NDA LO per the standard assay procedure described in CCP-TP-166.

4.5.9 Verify all required information is documented on Attachment 1.

NOTE

For Calibration Verification, accuracy SHALL **NOT** exceed +/- 30 percent. The required precision is reported as %RSD **AND** SHALL **NOT** exceed the values listed in Table 1.

4.5.10 Compare the mean measured value for Pu-240 Equivalent Mass and Pu-239 Effective Mass for the replicate measurements to the tag value for the source(s), **AND** indicate on Attachment 2.

4.5.11 Print name, sign, and date Attachment 1 and Attachment 2.

4.5.12 Provide electronic Calibration Data Files, Attachment 1, and Attachment 2 to the NDA LO for review.

NDA LO

NOTE

Signature by reviewer indicates all calculations have been validated.

4.5.13 Review Attachment 1 and Attachment 2 **AND** print name, sign, and date.

4.5.14 Provide electronic Calibration Data Files, Attachment 1, and Attachment 2 to the NDA EA.

NDA EA

4.5.15 Document the verification results in a Calibration Verification Report.

4.5.16 Transmit Calibration Data Files, Attachment 1 and Attachment 2, **AND** the Calibration Verification Report to the Facility Records Custodian.

4.6 Preparation of the Calibration and Validation Report

4.6.1 Upon completion of the DWAS IPAN calibrations, prepare a Calibration and Validation Report to include the following:

[A] Confirmation measurement results including a demonstration of precision and accuracy.

[B] All QA measurement control results and plots.

[C] Calibration and measurement results.

[D] Lower Limit of Detection (LLD) results.

[E] Identification of the number of replicates and types of matrices.

4.6.2 Print name, sign, and date the Calibration and Validation Report.

4.6.3 Transmit the Calibration and Validation Report to the Facility Records Custodian.

Facility Records Custodian

4.6.4 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 and Validation Report to include the following:

- Final Reports from Instrument Performance Background Measurements (from CCP-TP-166)
- Final Reports from Instrument Performance Source Measurements (from CCP-TP-166)

[B] Calibration Log Sheet(s), as required

- Calibration
- Validation
- Verification
- Confirmation

[C] Calibration Verification Report, as required

[D] Calibration Verification Sheet

Attachment 1 – Calibration Log Sheet

Calibration type Calibration/Validation/Verification/Confirmation _____
Facility _____
Source ID _____
Matrix ID (e.g.NDA-5183-001) _____
Gross Weight (kg) _____
Tare Weight (kg) _____
Net Weight (kg) _____

Perform measurements per CCP-TP-166. Complete the following information associated with each measurement. Initial each run for the data acquired.

Position #	Radius (in)	Height (in)	Database	Run Number	Matrix ID	QA Check	Initial
Background Check							
Instrument Performance Source Measurement							
NSBKG							
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
Calibration Confirmation/Verification	%RSD =	Accuracy = %R					
Comments							

Prepared by: Name/Title _____ Date _____

Reviewed by: Name/Title _____ Date _____

Attachment 2 – Calibration Verification Sheet (Example)

MCS

Calibration Verification

Site: Foster Wheeler, Melton Valley, TRU Facility
Facility: DWAS/SGS
Date: 1/15/2007

Passive Mode Calibration Verification

* (Pass/Fail) **PASS**

Source Location:	Tube 3, H 15 inches	
Drum ID:	NDA-5183-001	
Cf252 Source ID:	Isotope Products S/N C5-926	
Certification Date:	6/1/2005	
Measurement Date:	1/15/2007	
Elapsed Time (years)	1.62	
Initial Cf252 Cert Value	23810.0	**n/s Cf252**
Decayed Cf252 Value	15572.9	**n/s Cf252**
System Conversion factor	0.00168	**g Pu240/n/s Cf252**
Tag Value +30%	34.011	
Tag gram equivalent Pu240	26.162	**g/Pu240**
Tag Value -30%	18.314	
Mean Measured Value	24.224	**g/Pu240**
%R	92.59	
%RSD	0.30	

Measured Pu240 Effective Mass	**(g Pu240)**	**P file**
Replicate #1	24.13538	070115.P3
Replicate #2	24.28365	070115.P4
Replicate #3	24.25412	070115.P5

Active Mode Calibration Verification

* (Pass/Fail) **PASS**

Source Location:	Tube 3, H 15 inches	
Drum ID:	NDA-5183-001	
U238 Source ID:	PSC #8	
Certification Value	428.38	**g U238**
U235 gram value (g Total U X .002)	0.858	**g U235**
Pu239 effective Mass (g U235 /1.5)	0.572	**g Pu239**
Tag Value +30%	0.641	
Tag Self-Absorption Corrected Mass (g Pu239/1.16)	0.493	**g Pu239**
Tag Value -30%	0.345	
Mean Measured Value	0.494	**g Pu239**
%R	100.09	
%RSD	1.46	

Measured Pu239 Effective Mass	**(g Pu239)**	**P File**
Replicate #1	0.48996	070115.P3
Replicate #2	0.5018872	070115.P4
Replicate #3	0.4889399	070115.P5

*** Acceptance Criteria:** Reference DOE/WIPP-02-3122, Rev 4.0, Contact-Handled Transuranic Waste Acceptance Criteria

%R for a Non-interfering Matrix (Mean/Tag): +/-30%

%RSD for a Non-interfering Matrix:

Number of Replicates	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Max %RSD	1.8	6.6	10.0	12.3	14.0	15.2	16.2	17.1	17.7	18.3	18.8	19.3	19.7	20.0

*The values listed are derived from the measured standard deviation of the replicate measurements using $\frac{s}{\mu} * 100\% < \sqrt{\frac{(0.292) * \chi^2_{0.05,n-1}}{n-1}} * 100\%$ where s is the measured standard deviation, n is the number of replicates, μ is the true value, $\chi^2_{0.05,n-1}$ is the critical value for the upper 5% tail of a one sided chi-squared distribution with n-1 degrees of freedom, and 0.292 corresponds to a 95% upper confidence bound on the true system precision limit of 29.2%.

Comments: 240 Pu above action limit

Generated by: (Print/Sign/Date) Deborah Satterfield 01/15/07

Reviewed by: (Print/Sign/Date) Barry Smith 01/15/07