

# CCP-TP-192

Revision 1

## CCP

# Box Neutron Assay System (BNAS) Calibration Procedure

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## 1.0 PURPOSE

The purpose of this procedure is to prescribe the method for calibrating the Box Neutron Assay System (BNAS). Gamma spectrometry data complementary to neutron data from the BNAS are acquired and analyzed using the Box Segmented Gamma System (BSGS). Contents of boxes of U.S. Department of Energy (DOE) waste containing transuranic (TRU) and other radioisotopes are characterized using combined results from the BNAS and BSGS.

### 1.1 Scope

This procedure provides specific instructions for calibrating the BNAS to assay the radioactive content of waste containers.

## 2.0 REQUIREMENTS

### 2.1 References

#### Baseline Documents

- ASTM C1207, *Standard Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting*
- 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)
- Document No. 38852, *Savannah River Box Counter System Neutron Box Counter Module, Hardware Reference Manual*
- CCP-PO-003, *CCP Transuranic Authorized Methods For Payload Control (CCP CH-TRAMPAC)*
- CCP-PO-005, *CCP Conduct of Operations*
- CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*

#### 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-QP-011, *CCP Notebooks and Logbooks*
- CCP-QP-022, *CCP Software Quality Assurance Plan*
- CCP-TP-191, *CCP Box Neutron Assay System 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 Personnel working around the BNAS conveyor system, door closure, and moving equipment must observe all warning devices and postings.

2.3.2 Personnel working around the BNAS shall be aware of potential electrical hazards.

2.3.3 Personnel shall be aware of the precautions to be exercised when working with or around radioactive sources and waste materials. Such precautions shall be in accordance with Host site requirements.

2.3.4 Quality control measurements involve the use of radioactive sealed sources. The physical position of the sources within the matrix container must be preserved to ensure repeatability.

## 2.4 Prerequisite Actions

### 2.4.1 Equipment Bay

[A] Ensure all personnel barriers are secure.

- [B] Verify that NO Emergency Stops are activated.
- [C] Inspect equipment and moving parts to ensure they are clear of any unauthorized personnel or obstructions.
- [D] Inspect the conveyor system to ensure that the conveyor rollers have **NOT** shifted.

#### 2.4.2 Control Room

- [A] Ensure the Light Stacks are operational.
- [B] Ensure that the JSR-14 Shift Registers are turned to the ON position.
- [C] Ensure that the Programmable Logic Controller (PLC) cabinet is turned ON and operational (e.g., supplies system power, provides troubleshooting capability, etc.).

### 2.5 Equipment List

#### 2.5.1 Helium-3 (He-3) Detectors

#### 2.5.2 Conveyor mechanism (i.e., conveyor, drawbridge, etc.)

#### 2.5.3 Californium-252 (Cf-252) Add-A-Source (AAS)

#### 2.5.4 Electronics rack housing

- [A] JSR-14 Shift Registers
- [B] Personal computer with appropriate software and peripherals
- [C] Uninterruptible power supplies (UPSs)

#### 2.5.5 Five Light Stacks, (one in the control room and one on each exterior corner of the counter)

#### 2.5.6 Software

- [A] NDA2000, Waste Assay

2.6 Definitions

- 2.6.1 **Add-A-Source (AAS)** - A Cf-252 source ( $\sim 10^5$  n/s) introduced during an assay to correct the detected neutron signal for sample moderation and absorption. The AAS is stored in a shielded enclosure at the side of the BNAS. It is automatically controlled so it is never out of its shielded enclosure when a BNAS door is OPEN.

3.0 RESPONSIBILITIES

3.1 NDA Operator

3.1.1 Operates the BNAS in accordance with CCP-TP-191, *CCP Box Neutron Assay System Operating Procedure*.

3.1.2 Transfers calibration data to the nondestructive assay (NDA) Expert Analyst (EA).

3.2 NDA Expert Analyst (EA)

3.2.1 Provides the technical supervision for calibration of the system.

3.2.2 Reviews and approves the BNAS calibration data.

3.2.3 Performs calculations and updates parameters as required by CCP-QP-022, *CCP Software Quality Assurance Plan*.

3.2.4 Prepares Calibration and Validation Reports.

3.2.5 Reviews and approves the Calibration and Validation Reports.

3.3 NDA Services Technical Director

3.3.1 Reviews and approves the Calibration and Validation Reports.

3.4 Facility Records Custodian

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

#### 4.0 PROCEDURE

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##### **NOTE**

Should an abnormal situation occur that would cause a deviation from this procedure **AND** the abnormal condition can **NOT** be corrected **OR** resolved by this procedure, the NDA Operator will STOP BNAS operations, **AND** notify the NDA EA or NDA Lead Operator (LO). Any abnormality will be recorded in the NDA Operational Logbook in accordance with CCP-QP-011, *CCP Notebooks and Logbooks*.

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#### 4.1 Daily Quality Control (QC) Checks

4.1.1 Check that unauthorized personnel have left the equipment bay.

4.1.2 Perform daily QC Checks (QC Background Check and QC Calibration AAS Check) in accordance with CCP-TP-191.

#### 4.2 Neutron Coincidence Counter (NCC) Calibration (Mass Calibration)

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##### **NOTE**

The BNAS has been characterized at the factory for operation in the multiplicity mode with AAS correction for matrix effects. It can also be calibrated for analysis in NCC mode. This section provides the steps to calibrate the BNAS in NCC mode.

Calibration standards shall be traceable to a nationally accredited measurement program. Calibration is performed prior to assaying TRU waste items. Calibration is repeated if routine QC Checks, as described in CCP-TP-191, DO **NOT** meet the acceptance criteria **AND** the NDA EA judges that recalibration is required. In most cases, assay anomalies can be corrected without resorting to recalibration.

Calibrations shall be documented in the NDA Operational Logbook in accordance with CCP-QP-011. Calibration and confirmation results will be summarized and documented in a Calibration and Validation Report.

Calibrations are saved with the associated data files, and the date of the calibration is logged into the NDA Operational Logbook in accordance with CCP-QP-011.

A simple model will be used for the NCC Pu240<sub>eff</sub> mass to Reals rate. A separate AAS Calibration has been performed for each matrix type and container.

Upon completion of the calibration measurements and the insertion of the results into the appropriate parameter files, an NDA EA will approve the calibration parameters.

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##### **NOTE**

Homogeneous solids (S3000) and debris waste (S5000) will be assayed in the BNAS. Matrix corrections will be performed using the AAS feature of the BNAS. The corrections are described in the AAS Calibration section of this procedure.

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**NOTE**

The NDA Operator will perform calibrations as directed by the NDA EA.

The calibration parameters may be computed manually by the NDA EA and inserted into the appropriate fields of the NDA2000 Software in lieu of the steps indicated in Section 4.2.1.

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4.2.1 Prior to initiating the calibration measurements, create **OR** edit the Pu or Cf-252 Declaration as follows:

[A] Double-click on the NDA2000 Setup icon on the Desktop.

[B] Select the Neutron tab on the NDA2000 Setup screen.

[C] Click on the PU Declaration (or Cf252 Declaration) button.

[D] From the PU Declaration (or Cf252 Declaration) screen, review the list of Previously Defined Pu Declaration, **AND** determine if the list must be modified to include any new calibration standards as follows:

[D.1] **IF NO** new standards are to be declared, **THEN** click on the Close button to exit the Declaration menu.

[D.2] **IF** new standards are to be declared, **THEN** click on the New button, **AND** enter the required information on the Declaration - Creating New Declaration screen.

(a) Click on the Save button after entering all the required information for the new standard.

(b) Exit the PU Declaration (or Cf252 Declaration) screen after all the new standards have been declared.

4.2.2 Perform an Environmental Background Check in accordance with CCP-TP-191.

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**NOTE**

Each standard should be measured for at least 3600 seconds. Standard measurements follow the same steps as item assay.

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4.2.3 Perform the Passive Mass Calibration Measurement as follows:

- [A] Load the calibration reference container with standards as directed by the NDA EA.
- [B] Ensure the calibration reference container is loaded onto the conveyor.
- [C] Click on the first toolbar icon (a Green light), **AND** select Passive Mass Calibration Measurement from the drop-down menu.
- [D] Click on the Start Assay button.
- [E] On the Item Information entry screen, enter any descriptive information appropriate for the measurement.
- [F] Click on Done to start the assay.
- [G] Ensure the Forklift Operator unloads the calibration reference container from the conveyor.

4.2.4 Repeat steps 4.2.3[A] through 4.2.3[G] for each calibration reference container to be counted.

4.2.5 Forward the results to the NDA EA for review and inclusion in the Calibration and Validation Report.

4.2.6 Double-click on the NDA2000 Calibration icon on the desktop, **AND** perform the following:

- [A] Set Select a Calibration to Mass.
- [B] From the Calibration Operations menu, select Perform Calibration, **OR** select the Perform Toolbar icon.

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4.2.7 Set Sample Type to the appropriate matrix form.

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**NOTE**

The NDA2000 Mass Calibration Wizard will guide the NDA Operator through the calibration process. An existing calibration can be edited from the NDA2000 Mass Calibration Wizard by locating it in the Mass Calibration drop-down menu and clicking on the Edit Existing Mass Calibration button.

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4.2.8 Start the NDA2000 Mass Calibration Wizard by clicking on the New Mass Calibration button.

[A] On the Calibration Description screen, enter the calibration description, **AND** click on the Next> button.

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**NOTE**

The NDA2000 Mass Calibration Wizard will next present the Pu240<sub>eff</sub> mass results for all the calibration measurements for the selected sample type. Using the horizontal bar will allow the NDA Operator to see all of the data for each measurement.

The NDA EA will identify which data points DO **NOT** fit the curve. Discordant data points will be addressed in the Calibration and Validation Report prepared by the NDA EA.

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4.2.9 Review the results.

4.2.10 Click on the left-most field on the screen to change each acceptable result from Do Not Use to Use From Count File to indicate that the data will be used in the calibration.

4.2.11 Click on the Next> button to bring up the Calibration Plot screen.

4.2.12 Verify that the data points on the plot fit the curve.

[A] **IF** a data point DOES **NOT** fit the curve, as identified by the NDA EA,  
**THEN** click on the Back button, **AND** mark the discordant data point as Do Not Use.

4.2.13 Click on the Next> button to return to the Calibration Plot screen, **AND** repeat step 4.2.12[A] until the data points are consistent.

4.2.14 Enter the following information based on direction from the NDA EA:

[A] Fit Model.

[B] Number of Coefficients, as appropriate.

4.2.15 Verify the mass limits span the  $\text{Pu240}_{\text{eff}}$  mass range of the standards used in the calibration.

4.2.16 Forward the calibration plot to the NDA EA for review and inclusion in the Calibration and Validation Report.

4.2.17 Click on Finish to accept and store the calibration.

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**NOTE**

Approval of the calibration will be performed by the NDA Operator with concurrence from the NDA EA.

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4.2.18 To approve the calibration for use **AND** to bring up the list of all Mass Calibrations, perform the following:

[A] Click on Approval from the Calibration Operations menu, **OR** click on the Approve toolbar icon.

4.2.19 Check “√” the box corresponding to that calibration in the Approval screen to approve the selected calibration.

4.2.20 Clear its check “√” box to withdraw approval of the selected calibration.

4.2.21 Click on the OK button when finished.

4.2.22 **IF** the calibration for a sample type is to be the default calibration, **THEN** select the Set Default function on the Calibration Operations drop-down menu screen.

4.2.23 Verify the settings in the Set Default screen are correct, **AND** click on the Next> button.

4.2.24 Select a new default Mass Calibration from the second Set Default screen.

4.2.25 Click on the Save button to set the default Mass Calibration, **AND** return to the previous dialog.

#### 4.3 Re-Normalization After Replacement of an Add-A-Source (AAS)

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##### NOTE

The AAS Calibration includes both volume perturbations and matrix perturbations. A Cf-252 source is placed inside a surrogate matrix container for the matrix perturbation measurement and in an empty container for the volume perturbation measurement. At least four distinct matrix loadings are analyzed in this fashion for each container type (i.e., 55-gallon drum, standard waste box [SWB] or standard large box [SLB]-2). Surrogate matrix containers are selected based on their hydrogen content, and the hydrogen content of the matrices spans the range expected in both the homogeneous solids and debris waste stream containers.

The AAS Calibration was performed at the factory and does **NOT** need to be repeated. The calibration will be re-normalized whenever the Cf-252 source is replaced.

The Cf-252 source used for the AAS matrix correction must be replaced when indicated by Quality Assurance (QA) results and confirmed by a Canberra Service Representative. Each new source must be obtained from a supplier maintaining a nationally accredited measurement program.

The NDA Operator will perform re-normalization under the direct guidance of the NDA EA.

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4.3.1 Ensure NDA QC Checks per CCP-TP-191 have been completed successfully during the current shift.

4.3.2 To re-establish the Reference Rate after the installation of a new Cf-252 source, perform the following for each container type (i.e., 55-gallon drum, SWB, or SLB-2) for which the BNAS is calibrated:

[A] Ensure the Forklift Operator loads an empty container onto the pallet.

[B] From the NDA2000 Operations Program, click on the first toolbar icon (a Green light), **AND** select Add-A-Source Perturbation from the drop-down menu.

- [C] On the Assay-Routine screen, ensure that the Container Type is set to the container type to be loaded.
- [D] Click on the Start Assay button.
- [E] On the Item Information screen, enter a unique item identification (ID), set the Declaration and any additional information on the measurement, such as Source ID, as prompted by the screen.
- [F] Click on the Done button to start the measurement.
- [G] **WHEN** the analysis is complete, **THEN** review the printout, **AND** when appropriate, provide it to the NDA EA.
- [H] Click on the NDA2000 Calibration icon on the desktop, **AND** set Select a Calibration to Add-A-Source.
- [I] Select Perform Calibration from the Calibration Operations drop-down menu **OR** click on the Perform Toolbar icon.
- [J] Select the container type counted **AND** click on the Edit Existing AAS Calibration button on the first Add-A-Source Calibration screen.
- [K] On the second AAS Calibration screen, click on the Calculate AAS Calibration Points tab.
- [L] Press the Add-A-Source Reference button.
  - [L.1] Select the measurement that was just performed.
  - [L.2] Click on the Replace Point button.
- [M] Repeat step 4.3.2[L] for each data point (matrix type).
- [N] Click on OK when finished.
- [O] Approve the Re-Normalized calibration, **AND** select it as the default.
- [P] Exit the Calibration program.
- [Q] Ensure the Forklift Operator unloads the empty container.

- [R] Repeat steps 4.3.2[A] through 4.3.2[Q] until data for each container type has been processed.

#### 4.4 Confirmation of Calibration

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##### **NOTE**

Calibration confirmation shall be performed after each calibration or recalibration using a non-interfering matrix. However, matrices representative of the calibration range may also be tested as directed by the EA.

Source strengths shall span the range of TRU waste loadings to be assayed with the instrument. Sources used for calibration shall **NOT** be used for confirmation.

The NDA EA will specify the container type and matrix, the standard IDs and locations, and the number of replicates.

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##### **NDA EA**

- 4.4.1 Provide the appropriate source/matrix configurations to the NDA Operator.
- 4.4.2 Verify that source standard(s) are traceable to a nationally accredited measurement program.

##### **NDA Operator**

- 4.4.3 Position the source(s) within the matrix, or request source loading and confirm loading has been correctly completed.
- 4.4.4 Ensure QC Checks per CCP-TP-191 have been completed successfully during the current shift.
- 4.4.5 Perform replicates as specified by the NDA EA or Lead Operator for each source/matrix configuration in accordance with CCP-TP-191.

##### **NDA EA**

- 4.4.6 Evaluate the confirmation measurement results using the calibration confirmation criteria specified in the following table taken from CCP-PO-002, *CCP Transuranic Waste Certification Plan* and using a percent recovery limit of  $\pm 30$  percent.

Table 1. Calibration Confirmation Criteria

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] **IF** the criteria are **NOT** met,  
**THEN** evaluate the cause and, if necessary, repeat the calibration confirmation.

4.4.7 Document the results of the calibration confirmation in the Calibration and Validation Report, as appropriate.

4.5 Preparation of the Calibration and Validation Report

**NDA EA**

4.5.1 Upon completion of the Neutron and Gamma Calibrations, prepare a Calibration and Validation Report to include the following elements:

- [A] Confirmation measurement results
- [B] Performance measurement control limits
- [C] Calibration measurement results
- [D] Weekly Interfering Matrix Standard Specifications and Limits
- [E] Lower Limit Detection and Minimum Detectable Concentrations (MDC) results for WIPP-reportable radionuclides
- [F] Demonstration of precision and accuracy
- [G] Justification of the number of replicates and types of matrices
- [H] Copies of the certificates of all radioactive sources used in the calibration and calibration confirmations
- [I] Reporting thresholds for reportable radionuclides that are not measured

4.5.2 Print name, sign, and date the Calibration and Validation Report as the preparer.

4.5.3 Forward the Calibration and Validation Report to another NDA EA or the NDA Services Technical Director for review and approval.

**Second NDA EA or NDA Services Technical Director**

4.5.4 Review the Calibration and Validation Report.

4.5.5 **IF** the Report is acceptable,  
**THEN** print name, sign, and date to approve, **AND** submit the Calibration and Validation Report to the Facility Records Custodian.

4.5.6 **IF** the Report is **NOT** acceptable,  
**THEN** return the Calibration and Validation Report to the first NDA EA with critique for revision.

**NDA EA**

[A] Revise the Calibration and Validation Report and proceed to step 4.5.2.

**Facility Records Custodian**

4.5.7 Transmit the Calibration and Validation Report in accordance with CCP-QP-008.

#### 4.6 Calibration Verification

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##### **NOTE**

Verification of the calibration 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.

Calibration Verification shall be performed after any of the following conditions have occurred and the problem (if any) has been corrected:

- Major system repairs and/or modifications
- Replacement of major system components, including detectors and supporting electronic components that have the capability to affect the measurement results
- Significant changes to the system's software
- Relocation of the system
- Failure of measurement controls

The NDA EA or NDA LO will specify the container and matrix, serial numbers and locations of the Pu standards, and the number of assay replicates.

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##### **NDA Operator**

- 4.6.1 Arrange the appropriate source/matrix configuration as directed by the NDA LO or NDA EA.
- 4.6.2 Verify the source standard(s) are traceable to a nationally accredited measurement program.
- 4.6.3 Document the configuration and the number of measurements to be performed in the NDA Operational Logbook in accordance with CCP-QP-011.
- 4.6.4 Position the source(s) within the container, or request source loading and confirm loading has been correctly completed.
- 4.6.5 Ensure QC Checks in accordance with CCP-TP-191 have been completed successfully during the current shift.

- 4.6.6 Perform the number of replicates specified by the NDA EA for each source/matrix configuration in accordance with CCP-TP-191.

**NDA EA**

- 4.6.7 Evaluate the verification measurement results using the calibration confirmation criteria specified in Table 1.
- 4.6.8 **IF** the criteria are **NOT** met,  
**THEN** evaluate the cause and, if necessary, repeat the calibration verification.

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**NOTE**

Supporting data may include QA Last Results Reports, Long Reports, and radioassay data sheets.

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- 4.6.9 Document the configuration and number of measurements to be performed, results of the calibration verification, and any supporting data as determined by the NDA EA in a Calibration Verification Report.
- 4.6.10 Submit the Calibration Verification Report to the Facility Records Custodian in accordance with CCP-QP-008.

**Facility Records Custodian**

- 4.6.11 Transmit the Calibration Verification Report 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/Non-Permanent:

[A] Calibration and Validation Report to include the following:

[A.1] QC Background Check (from CCP-TP-191)

[A.2] QC Calibration AAS Check (from CCP-TP-191)

[A.3] Supporting data included as appendices and/or attachments

[B] Calibration Verification Report to include the following:

[B.1] Supporting data included as appendices and/or attachments