

# CCP-TP-064

Revision 5

## CCP

# Calibrating the High Efficiency Neutron Counter Using NDA 2000

EFFECTIVE DATE: 11/27/2007

D. K. Ploetz

PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	10/11/2003	Initial Issue
1	03/24/2004	Updated Sections 2.0, 4.0, 5.0 and 6.0 to coincide with changes to CCP-TP-063, resulting from the LANL MSA / LRA and Dry-runs from December 2003 - February 2004.
2	05/14/2004	Incorporated CBFO Adequacy Review and Audit comment resolutions into Sections 2.0-7.0 by removing all references to Canberra's Calibration and Validation Plan. Section 1.0 revised to update the Purpose with respect to the range of plutonium. Inserted steps 5.6 and 6.6 for clarification with respect to the calibration confirmation criteria.
3	09/10/2004	Redefinition of SME, EA, and TS functions.
4	03/31/2006	Update of HENC Energy Calibration. The operating range did not change, only the units of presentation have been adjusted to be consistent with the Calibration Report. The energy calibration was adjusted to 0.075 kev/channel, and an upper energy level of approximately 1200 kev. Administrative change to adjust calibration range discussion under Purpose section to be consistent with discussion in Purpose section of CCP-TP-063.
5	11/27/2007	Revised to remove restriction on High Efficiency Neutron Counter (HENC) #2 use. Certification letter for Los Alamos National Laboratory (LANL) Central Characterization Project (CCP) issued on July 11, 2007 approving HENC #2 for certification measurements. Clarified calibration range discussion per the US Environmental Protection Agency's acceptance of the changes in the calibration as a Tier 1 change.

TABLE OF CONTENTS

1.0	PURPOSE.....	4
1.1	Scope.....	4
2.0	REQUIREMENTS.....	4
2.1	References.....	4
2.2	Training Requirements.....	5
2.3	Equipment List .....	5
2.4	Precautions and Limitations .....	6
2.5	Prerequisite Actions .....	7
2.6	Definitions .....	7
3.0	RESPONSIBILITIES.....	8
3.1	HENC Operator.....	8
3.2	HENC Expert Analyst (EA).....	8
3.3	HENC Subject Matter Expert (SME) .....	8
3.4	Facility Records Custodian .....	8
4.0	PROCEDURE.....	9
4.1	Pre-Calibration Checks .....	9
4.2	Neutron Calibration .....	10
4.3	Gamma Calibration .....	17
4.4	Preparation of the Calibration and Validation Report .....	25
4.5	Verification of Calibration .....	26
4.6	Confirmation of Calibration.....	28
5.0	RECORDS.....	29

## 1.0 PURPOSE

The purpose of this procedure is to prescribe the method for calibrating the High Efficiency Neutron Counters (HENCs). The HENCs include Add-A-Source (AAS) matrix correction and Gamma Spectrometry. Mobile Characterization Services (MCS) personnel are responsible for determining the radioactive content of waste containers intended for shipment to the Waste Isolation Pilot Plant (WIPP). MCS personnel are responsible for the calibration of the HENCs.

This procedure applies to the measurement of homogeneous solids and debris waste in TRU waste drums that are measured by the HENC #1 and HENC #2 Systems at Los Alamos National Laboratory (LANL). Both HENCs have a neutron calibration range from Lower Limit of Detection (LLD) to 16.28 grams (g) <sup>240</sup>Pu-effective for multiplying waste forms (i.e., predominantly <sup>239</sup>Pu materials). For non-multiplying waste forms (i.e., predominantly <sup>238</sup>Pu materials) the neutron calibration range extends from LLD to 35.0 grams of <sup>240</sup>Pu effective. The plutonium mass calibration range for the gamma component of the HENC is from the LLD to 217g of Weapons Grade Pu as limited by dead time. From a gamma measurement standpoint, the HENCs are limited to measuring drums with a bulk density range between 0.018 g/cubic centimeter (cc) and 2.1 g/cc.

Calibrations will be normalized to 100 percent Recovery (%R) and system correction factors shall be established and algorithms adjusted such that the accuracy is set equal to 100 percent.

### 1.1 Scope

This procedure provides specific instructions for calibrating the HENC to assay the radioactive content of waste containers. This procedure describes the calibration of both the Neutron and Gamma components of the HENC.

## 2.0 REQUIREMENTS

### 2.1 References

#### Baseline Documents

- ASTM C 1207, *Standard Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting*
- Canberra Industries, Inc. Publication, *High Efficiency Neutron Counter Technical Reference Manual*
- 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. 96179, Rev. A, *Model HE-WDAS High Efficiency Waste Drum Assay System w/Add-A-Source Option, 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*
- CCP-QP-011, *CCP Notebooks and Logbooks*
- CCP-QP-022, *CCP Software Quality Assurance Plan*

#### 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-063, *CCP Operating the High Efficiency Neutron Counter Using NDA2000*

## 2.2 Training Requirements

- 2.2.1 All 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 HENC with integral gamma ray detector, cadmium gamma ray filter, and the AAS option.
- 2.3.2 Canberra Neutron Multiplicity Counter, Model 2150 or equivalent.
- 2.3.3 Canberra Digital Signal Processor, Model 9660 or equivalent.

2.3.4 Software:

- NDA 2000, Waste Assay
- Genie 2000, Gamma Acquisition and Analysis
- Multi Group Analysis (MGA), Isotopics

2.3.5 Stack Indicator Lights (Red, Yellow, Blue, and Green).

2.3.6 Performance Check Drum and Check Source(s).

2.3.7 Matrix Drums for Calibration.

2.3.8 Pu Standards (traceable to the national standards database).

2.3.9 Gamma Line Sources (traceable to the national standards database).

2.4 Precautions and Limitations

2.4.1 The HENC will start without an audible warning. The Nondestructive Assay (NDA) Operator shall control access to the equipment. The following hazards are addressed in Safety Training for personnel that operate the HENC System:

- Liquid nitrogen
- Sealed radioactive sources
- Drums containing radioactive and hazardous materials
- Moving parts (e.g., shield doors, conveyors)

2.4.2 The Gamma Detector includes a Dewar flask that must be filled periodically with liquid nitrogen. Caution will be exercised to prevent injury from the extremely cold temperature.

2.4.3 Only authorized personnel will handle radioactive sources.

2.4.4 HENC Operators will **NOT** handle drums containing radioactive or hazardous materials.

2.4.5 The HENC contains heavy moving parts and the HENC Operator will ensure that there are no personnel in the Equipment Bay during normal system operations.

2.5 Prerequisite Actions

- 2.5.1 Verify the HENC has been started up in accordance with CCP-TP-063, *CCP Operating the High Efficiency Neutron Counter Using NDA2000*.

2.6 Definitions

- 2.6.1 **AAS** - A Cf-252 source ( $\sim 10^5$  neutrons/second) introduced during an assay to correct the detected Neutron signal for sample moderation. The AAS is stored in an enclosure and shielded on top of the HENC. It is automatically controlled so it is never exposed while a HENC door is open.
- 2.6.2 **Half-life** - The time necessary for the number of nuclei of a specific radioactive isotope to decay to one-half of its initial value.
- 2.6.3 **Lower Limit of Detection (LLD)** - That radioactivity level which, if present, yields a measured value greater than the critical level with 95 percent probability, where the critical level is that value which measurements of the background will exceed with 5 percent probability.

### 3.0 RESPONSIBILITIES

#### 3.1 HENC Operator

3.1.1 Responsible for the operation of the HENC Assay System.

3.1.2 Performs all procedural steps, unless otherwise stated.

3.1.3 Starts documentation for the HENC calibration records.

#### 3.2 HENC Expert Analyst (EA)

3.2.1 Provides the technical supervision for the operation and calibration of the system.

3.2.2 Provides guidance in the event of abnormal conditions.

3.2.3 Prepares the Calibration and Validation Report.

3.2.4 Reviews and approves the HENC calibration data.

#### 3.3 HENC Subject Matter Expert (SME)

3.3.1 Reviews and approves the Calibration and Validation Report.

#### 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

| HENC Operator

---

**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 HENC Operator will STOP HENC operations **AND** notify the HENC EA. Any abnormality will be recorded in the NDA Operational Logbook.

---

4.1 Pre-Calibration Checks

4.1.1 Check that all personnel have left the Equipment Bay.

4.1.2 Perform an initial Environmental Background Measurement in accordance with CCP-TP-063.

## 4.2 Neutron Calibration

---

### NOTE

Calibration standards shall be traceable to national standards database. Calibration is performed once prior to assaying TRU waste items and repeated at such time that routine performance checks, as described in CCP-TP-063 DO **NOT** meet the acceptance criteria.

Calibrations shall be documented in the NDA Operational Logbook. Calibration and verification results will be summarized and documented in a Calibration and Validation Report.

Calibrations shall be checked **AND**, if necessary, re-performed if any of the following conditions exist:

- Completion of 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
- After a measurement of the system's calibration or other test demonstrates that the system's response has changed significantly

Calibrations are saved with the associated data files, and the date of the calibration is logged into the NDA Operational Logbook.

A simple model will be used to describe this relationship. A separate AAS Calibration shall be performed for each sample type and container.

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

---

**NOTE**

The Gamma-Ray Spectrometer accomplishes two functions: isotopic and quantitative analysis. An Energy Calibration of the Gamma-Ray Spectrometer is required for the isotopic analysis. Both an Energy Calibration and an Efficiency Calibration of the Gamma-Ray Spectrometer are required for the quantitative analysis.

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

The Los Alamos National Laboratory Special Nuclear Material (LANL SNM) Custodian will oversee the SNM used for the Neutron Calibration of the HENC. The SNM Custodian will physically load the SNM sources into the matrix drums in accordance with the HENC EA instructions.

---

4.2.1 Mass Calibration

---

**NOTE**

The HENC Operator will perform calibrations under the direct guidance of the HENC EA.

The calibration parameters may be computed manually by the HENC EA and inserted into the appropriate fields of the NDA 2000 Software in lieu of step 4.2.1.

---

- [A] Prior to initiating the calibration measurements, create **OR** edit the Pu Declaration as follows:
  - [A.1] Double-click on the NDA 2000 SETUP icon on the Desktop.
  - [A.2] Select the NEUTRON tab on the NDA 2000 Setup screen.
  - [A.3] Click on the PU DECLARATION Button.

- [A.4] From the PU 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:
- (a) **IF NO** new standards are to be declared, **THEN** click on the CLOSE Button to exit the Declaration menu.
  - (b) **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.
    - (b.1) After entering all the required information for the new standard, click on the SAVE Button.
    - (b.2) Exit the PU DECLARATION screen after all the new standards have been declared.

- [B] Perform an Environmental Background Measurement in accordance with CCP-TP-063.

---

**NOTE**

Each reference item should be measured for at least 3600 seconds. Standards measurement follows the same steps as item assay. For more information on assaying items, see CCP-TP-063.

---

- [C] Perform the Passive Mass Calibration Measurement as follows:
- [C.1] Instruct the Forklift Operator to load a calibration Reference Drum onto the conveyor.
  - [C.2] Click on the first toolbar icon (a Green light) **AND** select PASSIVE MASS CALIBRATION MEASUREMENT from the drop-down menu.
  - [C.3] Click on the START ASSAY Button.
  - [C.4] On the Item Information entry screen, enter any descriptive information germane to the measurement.
  - [C.5] Click on DONE to start the assay.

- [C.6] Print the results at the end of the assay, **AND** forward to the HENC EA for review and inclusion in the Calibration and Validation Report.
- [C.7] Instruct the Forklift Operator to unload the calibration Reference Drum from the conveyor.
- [D] Repeat steps 4.2.1[C.1] through 4.2.1[C.7] for each calibration Reference Drum to be counted.
- [E] Double-click on the NDA 2000 CALIBRATION icon on the Desktop **AND** perform the following:
  - [E.1] Set Select a Calibration to MASS.
  - [E.2] From the Calibration Operations menu, select PERFORM CALIBRATION **OR** select the PERFORM TOOLBAR icon.
- [F] Set Sample Type to the appropriate matrix form.

---

**NOTE**

The NDA 2000 Mass Calibration Wizard will guide the HENC Operator through the calibration process. An existing calibration can be edited from the NDA 2000 Mass Calibration Wizard by locating it in the Mass Calibration drop-down menu and clicking on the Edit Existing Mass Calibration Button.

---

- [G] Start the NDA 2000 Mass Calibration Wizard by clicking on the NEW MASS CALIBRATION Button.
  - [G.1] On the Calibration Description screen, enter the calibration description **AND** click on the NEXT> Button.

---

**NOTE**

The NDA 2000 Mass Calibration Wizard will next present the Pu-240 Effective Mass results for all the calibration measurements for the selected sample type. Using the horizontal bar will allow the HENC Operator to see all of the data for each measurement.

The HENC 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 HENC EA.

---

- [H] Review the results.

- [I] 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.
- [J] Click on the NEXT> Button to bring up the Calibration Plot screen.
- [K] Verify that the data points on the plot fit the curve.
  - [K.1] **IF** a data point does **NOT** fit the curve, **THEN** click on the BACK Button **AND** mark the discordant data point as DO NOT USE.
- [L] Click on the NEXT> Button to return to the Calibration Plot screen **AND** repeat step 4.2.1[K] [K.1] until the data points are consistent.
- [M] Enter the following information based on direction from the HENC EA:
  - [M.1] Fit Model.
  - [M.2] Number of Coefficients, as appropriate.
- [N] Verify the mass limits span the Pu-240 Effective Mass range of the standards used in the calibration.
- [O] Click on the PRINT icon to print the calibration plot **AND** forward to the HENC EA for review and inclusion in the Calibration and Validation Report.
- [P] Click on FINISH to accept and store the calibration.
- [Q] To approve the calibration for use **AND** to bring up the list of all Mass Calibrations, perform the following:
  - [Q.1] Click on APPROVAL from the Calibration Operations menu **OR** Click on the APPROVE toolbar icon.
- [R] To approve the selected calibration, check (✓) the box corresponding to that calibration in the Approval screen.
- [S] To unapprove the selected calibration, clear its check (✓) box.
- [T] Click on the OK Button when finished.

- [U] **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.
- [V] Verify the settings in the SET DEFAULT screen are correct, **AND** click on the NEXT> Button.
- [W] Select a new default MASS CALIBRATION from the second Set Default screen.
- [X] Click on the SAVE Button to set the default MASS CALIBRATION, **AND** return to the previous dialog.

#### 4.2.2 Re-Normalization After Replacement Of The AAS

---

##### **NOTE**

The AAS Calibration includes both volume perturbations and AAS perturbations. A Cf-252 source is placed inside a surrogate matrix drum for the volume perturbation measurement, and external to the drum for the AAS perturbation measurement. At least four surrogate matrix drums are analyzed in this fashion. Surrogate matrix drums 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 drums. Once the AAS Calibration is accomplished, the software can interpolate to find the appropriate matrix correction factor for any hydrogen loading in the calibration range.

The AAS Calibration was performed at the factory and needs **NOT** to be repeated. The calibration will be Re-Normalized every time 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 by a Canberra Service Representative. Each new source must have a new certificate.

The HENC Operator will perform Re-Normalization under the direct guidance of the HENC EA.

---

- [A] To re-establish the Reference Rate after the installation of a new Cf-252 source, perform the following:
  - [A.1] Instruct the Forklift Operator to load an empty drum onto the conveyor.

- [A.2] Click on the first toolbar icon (a Green light) **AND** select ADD-A-SOURCE PERTURBATION from the drop-down menu.
- [B] On the Assay-Routine screen, verify the Container Type is set to "55 gallon drum".
- [C] Click on the START ASSAY Button.
- [D] On the Item Information screen, enter a unique Item ID, set the Declaration **AND** any additional information on the measurement, such as Source ID, as prompted by the screen.
- [E] Click on the DONE Button to start the measurement.
- [F] Print the results at the end of the assay, **AND** forward to the HENC EA.
- [G] Click on the NDA 2000 CALIBRATION icon on the Desktop **AND** set Select a Calibration to ADD-A-SOURCE.
- [H] Select PERFORM CALIBRATION from the Calibration Operations drop-down menu **OR** click on the PERFORM TOOLBAR icon.
- [I] Select the 55 GALLON DRUM Container **AND** click on the EDIT EXISTING AAS CALIBRATION Button on the first Add-A-Source Calibration screen.
- [J] On the second Add-A-Source Calibration screen, click on the CALCULATE AAS CALIBRATION POINTS tab.
- [K] Press the ADD-A-SOURCE REFERENCE ellipsis Button.
- [K.1] Select the measurement that was just performed.
- [K.2] Click on the REPLACE POINT Button.
- [L] Repeat step 4.2.2[K] for each data point (matrix type).
- [M] Click on OK when finished.
- [N] Approve the Re-Normalized calibration **AND** select it as the default.
- [O] Exit the Calibration program.

[P] Instruct the Forklift Operator to unload the empty drum from the conveyor.

#### 4.3 Gamma Calibration

---

##### NOTE

Calibration standards shall be traceable to national standards database. Calibration is performed once prior to assaying TRU waste items and repeated at such time that routine performance checks, as described in CCP-TP-063, DO **NOT** meet the acceptance criteria.

Calibrations shall be documented in the NDA Operational Logbook. Calibration and verification results will be summarized and documented in a Calibration and Validation Report.

Calibrations shall be checked **AND**, if necessary, re-performed if any of the following conditions exist:

- Completion of 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
- After a measurement of the system's calibration or other test demonstrates that the system's response has changed significantly

Calibrations are saved with the associated data files, and the date of the calibration is logged into the NDA Operational Logbook.

A Multi-curve Efficiency Calibration will be performed to establish the relationship between Matrix Density and Detector Efficiency.

Upon completion of the Gamma Calibration measurements and the insertion of the results into the appropriate parameter files, the HENC EA will approve the calibration parameters.

---

---

**NOTE**

The Gamma-Ray Spectrometer accomplishes two functions: isotopic analysis and quantitative analysis. An Energy Calibration of the Gamma-Ray Spectrometer is required for the isotopic analysis. Both an Energy Calibration and an Efficiency Calibration of the Gamma-Ray Spectrometer are required for quantitative analysis.

During operation, an audible warning will **NOT** sound before motion of the HENC starts. Access to the equipment bay shall be controlled by the HENC Operator during calibration operations. Personnel are **NOT** allowed in the equipment bay during calibration operations.

The HENC Operator will perform the calibration under the direct guidance of the HENC EA.

---

4.3.1 Gamma Energy Pre-Calibration

- [A] Double-click on the NDA 2000 SETUP icon **AND** perform the following:
  - [A.1] Select the GAMMA tab.
  - [A.2] Click on the CALIBRATION SOURCE CERTIFICATE EDITOR Button.
  - [A.3] Click on the NEW Button.
- [B] Enter certificate name (e.g., All Lines).
- [C] Using the "Certificates of Calibration" for all the line sources, sum the activities, **AND** enter the following:
  - [C.1] Nuclide
  - [C.2] Energy (e.g., 59.5 keV)
  - [C.3] Summed Rate (e.g., 65866.5  $\gamma$ /sec)
  - [C.4] % Uncert (e.g., 3.5%)
  - [C.5] Half-Life (e.g., 432.7 yrs)
  - [C.6] Uncert (e.g., 0.5 yrs)

- [D] Click on ADD ROW.
- [E] Repeat step 4.3.1[C] through 4.3.1[D] for each nuclide listed on the Certificate of Calibration, as needed.
- [F] Click on SAVE.

#### 4.3.2 Energy Calibration For Gamma-Ray Analysis

---

##### NOTE

The Certificates of Calibration for the six line sources used for calibration will be included in the Calibration and Validation Report.

The Gamma-Ray Energy Calibration is set from the Genie 2000 program and requires the use of one or more sources that emit Gamma-Rays with energies between approximately 50 keV and 1500 keV.

The Energy Calibration extends from a nominal range of 0 to 1200 keV and is set manually, using Genie 2000 software.

There are **NO** requirements to perform Neutron Calibrations before performing Gamma Calibrations. Gamma and Neutron Calibrations can be performed independently of each other at any time.

---

- [A] Position the mixed-nuclide Gamma sources in the HENC sample chamber near the Gamma-Ray Detector.
- [B] Exit all NDA 2000 programs, **AND** start the Genie 2000 Gamma Acquisition and Analysis Program.
- [C] Select OPEN DATASOURCE from the File drop-down menu, **AND** perform the following:
  - [C.1] Click on the DETECTOR SOURCE Button.
  - [C.2] Select BEGE 1 as the Datasource, **AND** click on the OPEN Button.
- [D] Adjust the Pole/Zero as necessary per the Genie 2000 Manual, **THEN** adjust the other instrument settings to produce 16 k spectra of approximately 0.075 keV/channel with an approximate Zero offset.
- [E] Acquire a spectrum of the reference sources for at least 300 seconds.

- [F] Select "SETUP ...", from the Calibrate drop-down menu, **AND** establish the following settings:
  - [F.1] Energy Units set to "keV".
  - [F.2] Tolerance Units set to "Energy".
  - [F.3] Analysis Sequence Description set to "Energy Calibration Report".
  - [F.4] Calibration settings:
    - (a) Energy Cal set to "1.5 keV".
    - (b) Eff Match set to "1.0 keV".
  - [F.5] Tail Curves set to NONE.
  - [F.6] Continuum set to STEP.
  - [F.7] Channels set to "4".
- [G] Click on the OK Button to accept the settings.
- [H] Select ENERGY ONLY CALIBRATION from the Calibrate drop-down menu, **AND** perform the following:
  - [H.1] Position the cursor on the 121.8 keV spectral peak, **AND** click on the CURSOR Button to automatically determine the Peak Channel.
  - [H.2] Enter the energy value in the Energy box, **AND** click on the ACCEPT Button.
  - [H.3] Repeat steps 4.3.2[H][H.1] and 4.3.2[H][H.2] for the 1112.0 keV spectral peak.
  - [H.4] Click on the OK Button to accept the Two-Point Calibration.
- [I] Select ENERGY FULL>BY CERTIFICATE FILE, from the Calibrate drop-down menu, **AND** perform the following:
  - [I.1] Select the ALL LINES Certificate File created in step 4.3.1.
  - [I.2] Click on the AUTO Button.

- [I.3] Click on the SHOW Button, **AND** set the ORDER OF THE POLYNOMIAL to 2.

#### **HENC EA**

- [I.4] Review both the Energy and Shape Curves to determine if any discordant points exist.
- [I.5] Delete any points that are discordant, for assignable cause, **AND** annotate the deletions and causes in the Calibration and Validation Report.

#### **HENC Operator**

- [I.6] Print the amended Energy and Shape Curves **AND** the Calibration and Validation Report.
- [I.7] Click on the USE-RESULTS Button to accept the Energy and Shape Calibration Curves.
- [I.8] Click on the OK Button to finish the Energy Calibration.
- [J] Select SAVE, from the File drop-down menu, to preserve the Datasource's new Energy Calibration.

#### 4.3.3 Reference Peak Calibration for Gamma-Ray Analysis

- [A] Remove the mixed-nuclide Gamma sources from the HENC sample chamber.
- [B] Exit all NDA 2000 programs, **AND** start the Genie 2000 Gamma Acquisition and Analysis Program.
- [C] Select OPEN DATASOURCE, from the File drop-down menu, **AND** perform the following:
  - [C.1] Click on the DETECTOR SOURCE Button.
  - [C.2] Select BEGE 1 as the Datasource, **AND** click on the OPEN Button.
- [D] Set the Pulser Amplitude to produce a spectral peak at approximately 1150 keV.
- [E] Set the Pulse Rate to approximately 100 Hz.

- [F] Acquire a spectrum of the Pulser peak alone, for at least 100 seconds.
- [G] Select PEAK LOCATE>UNIDENTIFIED 2<sup>nd</sup>, from the Analyze drop-down menu, **AND** perform the following:
  - [G.1] Check (✓) the “Generate Report” box.
  - [G.2] Click on the EXECUTE Button.
- [H] Select PEAK AREA>SUM/NON-LINEAR LSQ FIT...” from the Analyze drop-down menu, **AND** perform the following:
  - [H.1] Check (✓) the Generate Report box.
  - [H.2] Click on the EXECUTE Button.
- [I] Select PRINT REPORT WINDOW from the File drop-down menu to generate a printout.
- [J] Forward the printout to the HENC EA for review **AND** inclusion in the Calibration and Validation Report.
- [K] Double-click on the NDA 2000 REFERENCE PEAK CALIBRATION icon on the computer Desktop.
- [L] Using the File menu or the OPEN toolbar icon, OPEN the Gamma detector as a DATASOURCE.
- [M] From the Reference Peak Calibration Program, perform the following:
  - [M.1] Select the appropriate Spectral Peak, **AND** designate it as the Reference Peak.
  - [M.2] Select the Reference Peak from the peaks in the lower grid, **AND** check (✓) the Select As Reference box.
  - [M.3] Select PULSER as the Source Type.
- [N] From the File drop-down menu, perform the following:
  - [N.1] Save the Reference Peak information in the Detector Datasource.

[N.2] CLOSE the NDA 2000 Reference Peak Calibration Program.

#### 4.3.4 Gamma-Ray Efficiency Calibration

- [A] Remove all Gamma sources **NOT** used in the calibration from the HENC sample chamber.
- [B] Verify all Calibration sources are traceable to the national standards database.
- [C] Provide five matrix drums that have been prepared in accordance with the HENC EA instructions.

---

#### NOTE

Up to 45 measurement data points can be used for the Efficiency Calibration. A uniform distribution of source material is approximated by using line sources (sources with uniformly deposited radioactivity along the length of the rod) placed vertically in the drum at specific radial distances such that when the drum is rotated the radiation response seen by the detector is uniform. The system will automatically perform a full efficiency calibration and display a plot of the results.

---

- [D] Assemble a Reference Drum, **AND** insert the Calibration Sources into their assigned tube positions in accordance with the HENC EA instructions.
- [E] Instruct the Forklift Operator to load the Reference Drum onto the conveyor.
- [F] Double-click on the NDA 2000 OPERATIONS icon on the computer Desktop, **AND** perform the following:
  - [F.1] Click on the first toolbar icon (a Green light), **AND** select GAMMA EFFICIENCY CALIBRATION from the Count Type drop-down list.
  - [F.2] On the Assay - Routine screen, perform the following:
    - (a) Enter the Container Type.
    - (b) Set the Gamma Preset Counting Time.
  - [F.3] On the Item Identification screen, enter the following:
    - (a) Item ID.

- (b) For Description 1, enter the Matrix Drum ID.
  - (c) For Description 2, enter Reference Source ID Number(s).
  - (d) For Location, enter MCS HENC #1, LANL TA-54.
  - (e) Select the appropriate Matrix Type.
  - (f) Drum Gross Weight.
  - (g) For Sample Type, select NONE.
  - (h) Select the appropriate Declaration.
  - (i) Click on the DONE Button to start the measurement.
- [G] Upon completion of the measurement, forward the printout to the HENC EA for review and inclusion into the Calibration and Validation Report.
- [H] Instruct the Forklift Operator to remove the Reference Drum from the conveyor.
- [I] Repeat steps 4.3.4[D] through 4.3.4[H] for all Reference Drums to be measured.
- [J] CLOSE the NDA 2000 Operations Program.
- [K] Double-click on the NDA 2000 CALIBRATIONS icon on the computer Desktop, **AND** perform the following:
- [K.1] Select EFFICIENCY from the Select A Calibration drop-down menu.
  - [K.2] Select PERFORM CALIBRATION from the Calibration Operations drop-down menu, **OR** click on the PERFORM... icon.
  - [K.3] Select 55 GALLON DRUM from the drop-down list, **AND** click on the NEW EFFICIENCY CALIBRATION Button.
  - [K.4] Enter an appropriate Calibration Description, **AND** click on the NEXT Button.

- [K.5] On the Counts screen, check (√) all the relevant Efficiency Measurements, **AND** click on the NEXT Button.
- [L] Examine the plot, **AND** identify any discordant data points.
- [M] Click on the OVERPLOT RADIO Button to display multiple Calibration Curves.
  - [M.1] **IF** any discordant data points are noted **AND** can be deleted for assignable cause, **THEN** click on the BACK Button, **AND** uncheck them from the list.
  - [M.2] **IF** data points were deleted, **THEN** click on the NEXT Button, followed by the FINISH Button to save the new calibration.
- [N] Printout the calibration, **AND** forward it to the HENC EA for approval, designation as the Default Calibration for the appropriate matrices **AND** inclusion in the Calibration and Validation Report.

#### 4.4 Preparation of the Calibration and Validation Report

##### **HENC EA**

- 4.4.1 Upon completion of the Neutron and Gamma Calibrations, prepare a Calibration and Validation Report to include the following elements:
  - [A] Validation and confirmation measurement results
  - [B] All QA measurement control results and plots
  - [C] Calibration measurement results
  - [D] Control measurement results
  - [E] LLD results
  - [F] Demonstration of precision and accuracy
  - [G] Justification of the number of replicates and types of matrices

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

4.4.3 Forward the Calibration and Validation Report to the HENC SME for review and approval.

#### **HENC SME**

4.4.4 Review and approve the Calibration and Validation Report.

4.4.5 Forward the Calibration and Validation Report to the Facility Records Custodian for transmittal as a record in accordance with CCP-QP-008.

#### 4.5 Verification of Calibration

---

#### **NOTE**

Verification of the Calibration shall be 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 may be used.

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

- Completion of 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 the Quality Assurance (QA) Tests for peak centroid or resolution
- After a measurement of the system's calibration or other test demonstrates that the system's response has changed significantly

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

---

#### **HENC EA**

4.5.1 Obtain the appropriate source/non-interfering matrix (e.g., daily calibration, verification source and Zero-Matrix drum) configuration.

4.5.2 Verify the source standard(s) are traceable to the National Institute of Science and Technology (NIST), the New Brunswick Laboratory (NBL) or from the supplier(s) maintaining measurement systems traceable to the national standards database.

4.5.3 Position the source(s) within the matrix.

#### **HENC Operator(s)**

4.5.4 Perform background **AND** source instrument performance checks per CCP-TP-063.

4.5.5 Perform replicates for each source/matrix configuration per CCP-TP-063, as directed by the HENC EA.

#### **HENC EA**

4.5.6 Evaluate the verification measurement results using the calibration confirmation criteria specified in CCP-PO-002, *CCP Transuranic Waste Certification Plan*.

[A] **IF** the criteria are met,  
**THEN** the calibration is verified and **NO** further action is required.

[A.1] Document the calibration verification.

[A.2] Forward the Calibration Verification Report to the Facility Records Custodian in accordance with CCP-QP-008.

[B] **IF** the criteria are **NOT** met,  
**THEN** evaluate the cause.

---

4.6 Confirmation of Calibration

---

**NOTE**

Calibration confirmation shall be performed after each calibration or re-calibration using a non-interfering matrix. Typically, an empty drum is used for the confirmation measurements. However, matrices representative of the calibration range may also be tested as required. The confirmation shall apply to both the Neutron and Gamma Calibrations.

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

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

---

**HENC EA**

4.6.1 Obtain the appropriate source/matrix configurations.

4.6.2 Verify the source standard(s) are traceable to NIST, the NBL, or from the supplier(s) maintaining measurement systems traceable to the national standards database (The source standards will be different than the calibration sources).

4.6.3 Position the source(s) within the matrix.

**HENC Operator(s)**

4.6.4 Perform background **AND** source instrument performance checks per CCP-TP-063.

4.6.5 Perform replicates for each source/matrix configuration per CCP-TP-063, as directed by the HENC EA.

**HENC EA**

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

[A] **IF** the criteria are met,  
**THEN** the calibration is confirmed and **NO** further action is required.

[B] **IF** the criteria are **NOT** met,  
**THEN** evaluate the cause.

## 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
- [B] Raw Data (CD(s) - Primary/Backup)
- [C] Calibration Verification Report, as required