



# Standard Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting<sup>1</sup>

This standard is issued under the fixed designation C1207; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes the nondestructive assay of scrap or waste for plutonium content using passive thermal-neutron coincidence counting. This test method provides rapid results and can be applied to a variety of carefully sorted materials in containers as large as several thousand liters in volume. The test method applies to measurements of  $^{238}\text{Pu}$ ,  $^{240}\text{Pu}$ , and  $^{242}\text{Pu}$  and has been used to assay items whose total plutonium content ranges from 10 mg to 6 kg (1).<sup>2</sup>

1.2 This test method requires knowledge of the relative abundances of the Pu isotopes to determine the total Pu mass (Test Method C1030).

1.3 This test method may not be applicable to the assay of scrap or waste containing other spontaneously fissioning nuclides.

1.3.1 This test method may give biased results for measurements of containers that include large amounts of hydrogenous materials.

1.3.2 The techniques described in this test method have been applied to materials other than scrap and waste (2, 3).

1.4 This test method assumes the use of shift-register-based coincidence technology (4).

1.5 Several other techniques that are often encountered in association with passive neutron coincidence counting exist. These include neutron multiplicity counting (5, 6, Test Method C1500), add-a-source analysis for matrix correction (7), flux probes also for matrix compensation, cosmic-ray rejection (8) to improve precision close to the detection limit, and alternative data collection electronics such as list mode data acquisition. Passive neutron coincidence counting may also be combined with certain active interrogation schemes as in Test Methods C1316 and C1493. Discussions of these established techniques are not included in this method.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

C986 Guide for Developing Training Programs in the Nuclear Fuel Cycle (Withdrawn 2001)<sup>4</sup>

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1133 Test Method for Nondestructive Assay of Special Nuclear Material in Low-Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry

C1316 Test Method for Nondestructive Assay of Nuclear Material in Scrap and Waste by Passive-Active Neutron Counting Using  $^{252}\text{Cf}$  Shuffler

C1458 Test Method for Nondestructive Assay of Plutonium, Tritium and  $^{241}\text{Am}$  by Calorimetric Assay

C1490 Guide for the Selection, Training and Qualification of Nondestructive Assay (NDA) Personnel

C1493 Test Method for Non-Destructive Assay of Nuclear Material in Waste by Passive and Active Neutron Counting Using a Differential Die-Away System

C1500 Test Method for Nondestructive Assay of Plutonium

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.10 on Non Destructive Assay.

Current edition approved June 1, 2010. Published July 2010. Originally approved in 1991. Last previous edition approved in 2003 as C1207 – 03. DOI: 10.1520/C1207-10.

<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

by Passive Neutron Multiplicity Counting  
**C1592** Guide for Nondestructive Assay Measurements  
**C1673** Terminology of C26.10 Nondestructive Assay Methods

2.2 *ANSI Standards*:<sup>5</sup>

**ANSI 15.20** Guide to Calibrating Nondestructive Assay Systems

**ANSI 15.36** Nondestructive Assay Measurement Control and Assurance

### 3. Terminology

3.1 Refer to Terminology **C1673** for definitions used in this test method.

### 4. Summary of Test Method

4.1 The even mass isotopes of Pu fission spontaneously. On the average, two or more prompt neutrons are emitted per fission event. The number of time correlated or coincident neutrons detected by the instrument is related to the effective mass of <sup>240</sup>Pu,  $m_{eff}$ , present in the time. The effective <sup>240</sup>Pu mass is a weighted sum of the even mass isotopes of Pu in the assay item. The total Pu mass is determined from the known plutonium isotopic ratios and the measured quantity  $m_{eff}$ .

4.2 The shift register technology is intended to correct for the effects of *Accidental* neutron coincidences which result from the registration of neutrons in the coincidence gate which are not correlated in time to the neutron which triggered the inspection of the gate.

4.3 Other factors which may affect the assay are neutron self multiplication, matrix components with large ( $\alpha$ , n) reaction rates, neutron absorbers, or moderators. Corrections for these effects are often not possible from the measurement data alone, consequently assay items are commonly sorted into material categories or additional information is sometimes used.

4.4 Corrections are typically made for electronic deadtime and neutron background.

4.5 Calibrations are typically based on measurements of well documented and appropriate reference materials. Modeling based on knowledge of the instrument design and the physical principles of neutron interactions may also be applied.

4.6 This method includes measurement control tests to verify reliable and stable performance of the instrument.

### 5. Significance and Use

5.1 This test method is useful for determining the plutonium content of scrap and waste in containers ranging from small cans with volumes of the order of a mL to crates and boxes of several thousand liters in volume. A common application would be to 208-L (55-gal) drums. Total Pu content ranges from 10 mg to 6 kg (**1**). The upper limit may be restricted depending on specific matrix, calibration material, criticality safety, or counting equipment considerations.

5.2 This test method is applicable for U.S. Department of Energy shipper/receiver confirmatory measurements (**9**), nuclear material diversion detection, and International Atomic Energy Agency attributes measurements (**10**).

5.3 This test method should be used in conjunction with a scrap and waste management plan that segregates scrap and waste assay items into material categories according to some or all of the following criteria: bulk density, the chemical forms of the plutonium and the matrix, americium to plutonium isotopic ratio, and hydrogen content. Packaging for each category should be uniform with respect to size, shape, and composition of the container. Each material category might require calibration standards and may have different Pu mass limits.

5.4 Bias in passive neutron coincidence measurements is related to item size and density, the homogeneity and composition of the matrix, and the quantity and distribution of the nuclear material. The precision of the measurement results is related to the quantity of nuclear material, the ( $\alpha$ ,n) reaction rate, and the count time of the measurement.

5.4.1 For both benign matrix and matrix specific measurements, the method assumes the calibration reference materials match the items to be measured with respect to the homogeneity and composition of the matrix, the neutron moderator and absorber content, and the quantity of nuclear material, to the extent they affect the measurement.

5.4.2 Measurements of smaller containers containing scrap and waste are generally more accurate than measurements of larger items.

5.4.3 It is recommended that where feasible measurements be made on items with homogeneous contents. Heterogeneity in the distribution of nuclear material, neutron moderators, and neutron absorbers have the potential to cause biased results.

5.5 The coincident neutron production rates measured by this test method are related to the mass of the even number isotopes of plutonium. If the relative abundances of these isotopes are not accurately known, biases in the total Pu assay value will result.

5.6 Typical count times are in the range of 300 to 3600 s.

5.7 Reliable results from the application of this method require training of the personnel who package the scrap and waste prior to measurement and of personnel who perform the measurements. Training guidance is available from ANSI 15.20, Guides **C986**, **C1009**, **C1068**, and **C1490**.

### 6. Interferences

6.1 Conditions affecting measurement uncertainty include neutron background, moderators, multiplication, ( $\alpha$ , n) rate, absorbers, matrix and nuclear material heterogeneity, and other sources of coincident neutrons. It is usually not possible to detect these problems or to calculate corrections for these effects from the measurement data alone. Consequently, assay items are sorted into material categories defined on the basis of these effects.

6.2 Neutron background levels from external sources should be kept as low and as constant as practical. Corrections can be

<sup>5</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

made for the effects of high-neutron background levels, but these will adversely affect measurement precision and detection limits.

6.3 Neutron moderation by low atomic mass materials will not only increase thermal-neutron absorption effects, but will also increase multiplication effects. Consequently, the measured neutron rates may be either smaller or larger than those for a nonmoderating matrix. Hydrogenous matrices contribute the most to this effect (11).

6.4 Both spontaneous and induced fissions produce coincident neutrons. The instrument, however, cannot distinguish between them. Three factors that strongly affect the degree of multiplication are the mass of fissile material, its density, and its geometry. Increases in mass that are not accompanied by changes in either density or geometry will result in predictable multiplication increases that can be incorporated into the calibration function. Localized increases in nuclear material density and/or changes in the geometry are likely to cause unknown changes in multiplication and measurement bias.

6.5 Neutrons from ( $\alpha$ , n) reactions are an interference bias source if they induce multiplication effects. In addition, ( $\alpha$ , n) neutrons can increase the Accidentals rate thereby affecting the statistical precision of the assay which is based on the net coincidence rate.

6.6 Biases may result from non-uniformity in the source distribution and heterogeneity in the matrix distribution.

6.7 Other spontaneous fission nuclides (for example, curium or californium) will increase the coincident neutron count rates, causing an overestimation of the plutonium content.

6.8 Cosmic rays, which are difficult to shield against, can produce coincident neutrons. Cosmic ray effects become larger for small quantities of Pu in the presence of large quantities of relatively high atomic number materials, for example, iron or lead are more prolific producers than cellulosic wastes (see 12.5).

## 7. Apparatus

7.1 *Counting Assembly*—See Fig. 1.

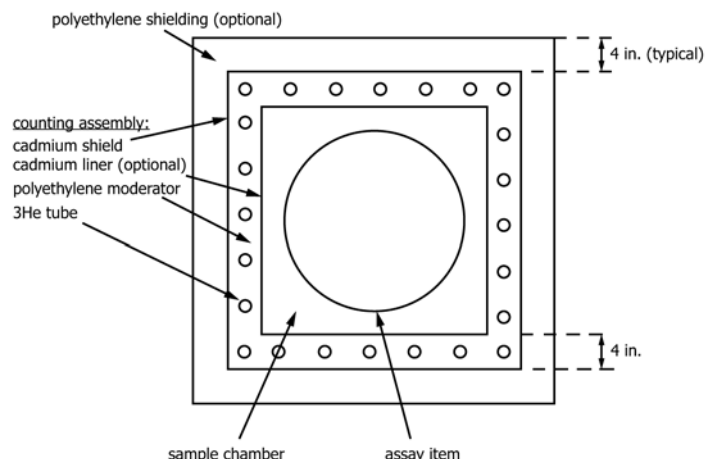


FIG. 1 A Cross-section View of a Typical Thermal-Neutron Coincidence Counter

7.1.1 The apparatus used in this test method can be obtained commercially. Specific applications may require customized design. The neutron detectors are usually  $^3\text{He}$  proportional counters embedded in polyethylene. The detection efficiency for neutrons of fission energy is typically at least 15 %. Larger detection efficiencies provide better precision and lower detection limits for a given count time. A short dieaway time is also important in that it allows a shorter gate width to be used which in turn helps control the Accidents. Ideally, the counter detection efficiency should vary less than 10 % over the item volume. The coincident response varies as the square of the detection efficiency.

7.1.2 Reproducible positioning of the item in the assay chamber is important for obtaining the best accuracy. This counting geometry should be maintained for the measurement of all reference materials and assay items. (See 11.7.)

7.1.3 A 0.4 mm to 1mm thick cadmium liner (12) is often installed on the inside surfaces of the counting chamber surrounding the assay item. This liner will reduce the dieaway time, decrease multiplication inside the item from returning neutrons and decrease the effects on the assay of neutron absorbers inside the item. The liner will also decrease neutron detection efficiency due to absorption of thermalized neutrons and may increase the cosmic ray spallation background. The final design may represent a compromise between multiple conflicting influences.

7.2 *Shielding*—The detector assembly is often surrounded by cadmium and an additional layer of hydrogenous material (see Fig. 1). Approximately 100 mm of polyethylene can reduce the neutron background in the assay chamber by approximately a factor of 10 (13).

7.3 *Electronics*—High-count-rate nuclear electronics provide a standard logic pulse from the  $^3\text{He}$  proportional counters. These pulses are processed by the shift-register coincidence technology.

7.4 Data acquisition and reduction can be facilitated by interfacing the instrument to a computer.

## 8. Hazards

8.1 *Safety Hazards*—Consult qualified professionals as needed.

8.1.1 Precautions should be taken to prevent inhalation, ingestion, or the spread of Pu contamination during waste or scrap handling operations. All containers should be surveyed on a regular basis with an appropriate monitoring device to verify their continued integrity.

8.1.2 Precautions should be taken to minimize personnel exposure to radiation.

8.1.3 Precautions should be taken regarding nuclear criticality, especially of unknown items. The measurement chamber approximates a reflecting geometry for fast neutrons. The assumption that waste is not of criticality concern is not recommended.

8.1.4 Counting chambers may contain a cadmium liner. Precautions should be taken to prevent the inhalation or ingestion of cadmium. It is a heavy metal poison. Cadmium shielding should be covered with nontoxic materials.

8.1.5 Precautions should be taken to avoid contact with high voltage. The  $^3\text{He}$  proportional counters require low current, high voltage, power supplies.

8.1.6 The weight of the instrument may exceed facility floor loading capacities. Check for adequate floor loading capacity before installation.

## 8.2 Technical Hazards:

8.2.1 Locate the instrument in an area of low-neutron background. Prohibit the movement of radioactive material in the vicinity of the instrument while a measurement is in progress.

8.2.2 Utilizing a measurement result outside of the calibration range should be carefully evaluated and, in general, is not recommended.

8.2.3 Utilizing a measurement result based on a calibration for a different material category should be carefully evaluated and, in general, is not recommended.

## 9. Instrument Preparation and Calibration

NOTE 1—Instrument preparation, determination of material categories, and calibration of passive neutron coincidence counters is discussed in the section below. Many details of these operations are site specific, depend on the matrix categories and nuclear materials to be measured, and should be evaluated by subject matter experts. Additional sources of information are Guide C1592 and ANSI 15.36.

### 9.1 Initial Preparation of Apparatus:

9.1.1 Locate the instrument in an area with the lowest practical neutron background. Prohibit the movement of radioactive material in the vicinity of the instrument while a measurement is in progress.

9.1.2 Perform the initial setup recommended by the system manufacturer in consultation with subject matter experts.

9.1.3 If the high-voltage plateau, die-away time, and dead-time correction coefficients were not supplied by the manufacturer, determine them. Consult an appropriate text on radiation detectors (14) or the manufacturer if assistance is needed and involve subject matter experts. Repeating these determinations can be a powerful check on the operational health of the instrument.

9.1.4 Set the gate length if it is a user adjustable feature. The optimum gate length for a wide range of count rates is about 1.257 times the die-away time (15). Low count rate applications sometimes benefit from longer gate lengths changing the gate length alters all calibrations. Whenever the gate length is changed, the instrument must be recalibrated.

9.1.5 Place the necessary cadmium liners in the assay chamber if it is a user adjustable feature. Very low gram quantity applications benefit from having no cadmium liner. Separate calibrations are required for each cadmium liner configuration.

9.1.6 Use a stable neutron source and refer to vendor's manuals to verify that the electronics are stable and operating properly.

9.1.6.1 Place a source of coincident neutrons, for example,  $^{252}\text{Cf}$  with an emission rate of  $\sim 5 \times 10^4 \text{ n.s}^{-1}$ , in the center of the counting chamber. Determine the Totals ( $T$ ), Reals ( $R$ ), and Accidentals ( $A$ ) neutron count rates from the accumulated quantities divided by the count time. A necessary but not sufficient indication of proper electronics operation is agree-

ment between  $A$  and the calculated quantity (calculated from the product of the square of the Totals rate and the gate width) within counting statistics or a predefined threshold.

9.1.6.2 Leaving the  $^{252}\text{Cf}$  neutron source inside the assay chamber, place a source of random neutrons, for example, americium-lithium with an emission rate of  $\sim 5 \times 10^4 \text{ n.s}^{-1}$ , in, or near, the counting chamber. Determine the Reals rates from the measured quantities for  $^{252}\text{Cf}$  with and without the random neutron source. The Reals rates should agree to within counting statistics for the two measurements (see 11.1).

9.1.6.3 Use these measurements as part of the measurement control data described in 10.1.

### 9.2 Determination of Material Categories for Required Calibrations:

9.2.1 Use this test method in conjunction with a scrap and waste management plan that segregates scrap and waste materials into categories with respect to the characteristics discussed in 5.3, and Sections 6 and 12. Packaging for each category defined should be uniform. Each material category will require a set of representative reference materials.

9.2.2 The material categories are normally one of three classifications: oxide, metal, or salt.

9.2.3 The effectiveness of the scrap and waste management plan and the validity of the resulting calibrations are best evaluated by the  $R/T$  ratio check described in Appendix X1.

### 9.3 Preparation and Characterization of Reference Materials (Guide C1128):

9.3.1 Calibration items should be as similar as possible to the assay items with respect to parameters such as size, shape, and composition which affect the measurement (see 5.3).

9.3.1.1 The Pu mass loadings should ideally span the range of loadings expected in the assay items and be adequate to define the shape of the calibration curve. Three to eight mass loadings are deemed suitable for each material category.

9.3.1.2 The Reals-to-Totals ratio, ( $R/T$ ), may be used as an indicator to determine whether the neutron emission characteristics of the measured item matches the reference materials. Reasonable agreement between the  $R/T$  ratios for the reference materials and assay items (defined by a facility-dependent evaluation for each material category) suggests that the reference material is appropriate. See Appendix X1 for more information.

9.3.2 For waste measurements of small gram quantities of plutonium, dilute the plutonium used in the reference materials sufficiently to eliminate multiplication effects.

9.3.3 The accuracy of the calibration items should be established; ideally by a technique that has significantly smaller measurement uncertainty than that desired for the coincidence counter results.

9.3.4 Permanently record the following information for each calibration item: packaging material(s), matrix, plutonium mass,  $m_{\text{eff}}$ , plutonium isotopic composition, and americium content with the date(s) measured.

### 9.4 Calibration Procedure—Use the following calibration procedure for each material category.

9.4.1 Calibration of a neutron coincidence counting instrument determines the relationship between the Reals count rate ( $R$ ) and the  $^{240}\text{Pu}$  effective mass,  $m_{\text{eff}}$ .



9.4.2 Measure each calibration item such that the measurement precision is substantially (for example, 3 to 5 times) better than that expected for assay items of similar Pu mass. See Section 10.2 for counting procedures and Section 11 for required calculations.

9.4.3 Choice of calibration functions will depend on the characteristics of the material category as indicated below.

9.4.3.1 Measurements of small quantities of Pu that exhibit no multiplication will normally show a linear relation of the form:

$$R = a_0 + a_1 m_{eff} \quad (1)$$

where  $a_1$  and  $a_0$  are coefficients determined by the fitting procedure.

9.4.3.2 Measurements of large quantities of Pu of consistent chemical form and item geometry, often show a calibration function of the form:

$$R = a_0 + a_1 m_{eff} + a_2 (m_{eff})^2 \quad (2)$$

here  $a_2$ ,  $a_1$ ,  $a_0$  are coefficients determined by the fitting procedure.

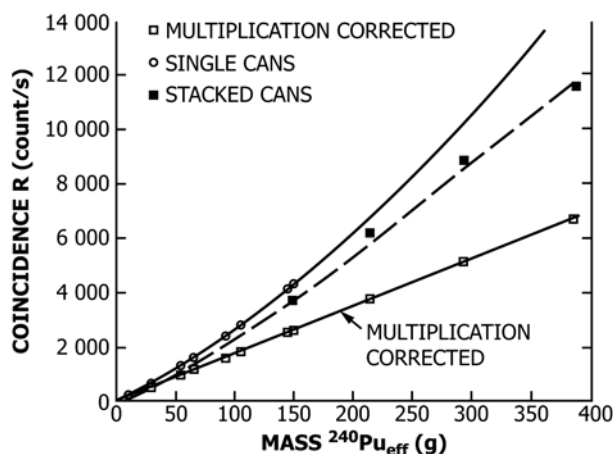
9.4.3.3 If the calibration is to be extrapolated to total Pu masses below the range of calibration, the parameterization may produce less bias if  $a_0$  is set to zero rather than fitted.

9.4.4 Record the allowed range of plutonium mass for the material category. The largest plutonium reference item typically places an upper limit on the assay range. Similarly, the lowest-valued plutonium reference item typically places a lower limit on the assay range. Utilizing a measurement result outside of the range of the calibration is not recommended.

9.4.5 Fig. 2 illustrates a problem that may occur when large plutonium mass items are simulated by stacking cans on top of each other. Because of geometric decoupling, self-multiplication is less than expected for a single can with the same high mass.

## 10. Procedure

NOTE 2—After calibration, the analytical procedure consists of measurements that demonstrate that the apparatus is calibrated and functioning



NOTE 1—Measured coincidence rate for two different measurement geometries (upper two curves) and multiplication corrected rate (bottom curve). Data for the curves was taken from Reference (16).

FIG. 2 Calibration Curves for Plutonium in a Neutron Coincidence Counter

properly (measurement control) and measurements of items with unknown Pu content.

10.1 *Measurement Control*—The need for adjustment of the instrument can be determined by measurement control procedures (17). Frequent measurement of the rates of a reference material should be used to validate proper instrument operation. If instrument malfunction is suspected, perform all measurement control tests (Section 9.1.6) to provide data helpful to analyze the condition of the measurement system (Sections 10.1.1 – 10.1.4). Maintain measurement control charts to archive and monitor measurement control results and to make decisions about the need for calibration or maintenance (Reference Guide C1210). If measurement control indicates the instrument response has changed, determine the cause of the change. Then it will be clear whether to repair the instrument or repeat the calibration procedure, or both.

10.1.1 Perform periodic background counts before the measurement of assay items. Changes in the  $R$  and  $T$  values from historical values should be investigated (18).

10.1.2 Perform periodic counts of a well-characterized item or reference material to verify the long-term stability of the instrument. Typical practice is a daily check, if the instrument is used daily. For less frequent use, typical practice is to perform an instrument check before and after each period of use. Agreement of the measurement value with its reference value, within control limits, indicates proper operation of the instrument. Low results may indicate that a detector or detector bank is not functioning. High results may indicate electrical noise.

10.1.2.1 The item being used for the instrument check must provide a consistent coincidence signal. Suitable items are a <sup>252</sup>Cf source corrected for radioactive decay (including allowance for <sup>250</sup>Cf where necessary), a reference material, or other stable source in which the material is fixed. Any characteristic which affects the Reals must not vary between measurements. Using a source in which the material is likely to change in some respect, such as bulk density, shape, or position of the material in the outer container, is not recommended.

10.1.3 Systematically perform replicate measurements of items to verify that the assumption of Poisson counting statistics is valid. This test might be done monthly or after each calibration. Statistical agreement between the standard deviation of the replicates and the uncertainty estimate based on counting statistics from each replicate indicates adequate stability of the instrument. Lack of agreement suggests background variations or electrical instabilities.

10.1.4 If measurement control criteria are passed, proceed to assays. If measurement control criteria fail, diagnose and correct the problem. Then proceed to setup, calibration, or repeat measurement control measurements.

### 10.2 Item Measurements:

10.2.1 If possible, center the assay item both vertically and horizontally in the counting chamber. This counting geometry should be maintained for all reference materials and assay items.

10.2.2 Count for the chosen count time.

10.2.3 When the count is complete, record, at a minimum, the assay item identifier, the accumulated counts in the Totals,

(R+A) and A scalars and the elapsed count time. For neutron coincidence counters under computer control, this information is recorded automatically.

10.2.4 Remove the assay item from the counting chamber.

10.2.5 Proceed to calculate the amount of Pu present in the assay item.

10.2.6 The following diagnostic tests are recommended for each measurement.

10.2.6.1 The Totals neutron count rate can be used to estimate the Accidentals rate as shown in 9.1.6.1. Lack of agreement within acceptable limits suggests a hardware failure in the coincidence circuitry or that the background neutron count rate changed significantly during the measurement.

10.2.6.2 Each measurement can be divided into several counting periods, and statistical tests can be performed that look for outliers in the individual counting periods (8). This “outlier” test reduces the effects of cosmic-ray background or of changing conditions during the measurement.

## 11. Calculation

NOTE 3—This section provides a summary of some of the main calculations. The calculations are typically performed by the system software rather than by the operator. The vendor should provide assurance that the calculations are correctly implemented in the software. The calculations follow the same general approach whether the results are used for calibration, measurement control, or determining the Pu content in an item.

11.1 Estimate the standard deviation of the Reals coincidence rate for a single measurement according to:

$$\sigma_R w \cdot \sqrt{[(R + 2 \cdot A)/t]} \quad (3)$$

The weighting factor  $w$ , approximately equal to 1.20, is a function of the detector parameters and the count rates, and is included because the (R+A) and A rates are correlated (4, 18). The estimate of the standard deviation of the totals rate is:

$$\sigma_T \sqrt{[T/t]} \quad (4)$$

11.2 *Dead-time Correlation*—Items with large quantities of Pu or materials with a large source of ( $\alpha$ ,  $n$ ) neutrons can produce high count rates. It is important to make a correction for rate related counting losses (15). The corresponding corrected count rates,  $R_c$  and  $T_c$  are typically calculated as follows:

$$R_c = R \cdot \exp[(a + b \cdot T)T] \quad (5)$$

$$T_c = T \cdot \exp[(a + b \cdot T)T/4] \quad (6)$$

where  $a$  and  $b$  are deadtime parameters (2).

11.2.1 The manufacturer should supply the deadtime parameters with the delivery of the instrument. They depend on the instrument design; for example, the number of amplifier-discriminators (19).

11.2.2 Standard error propagation formulae apply to estimate the random uncertainty from counting statistics.

11.3 *Background Correction*—Subtract the corresponding background rate from the measured quantities.

$$R' = R - R_b \quad (7)$$

$$T' = T - T_b \quad (8)$$

where:

$R_b$  = Reals rate for a blank item (but typically taken to be the rate with an empty chamber) and

$T_b$  = Totals rate for a blank item (but typically taken to be the rate with an empty chamber).

11.4 Determine  $m_{eff}$  from the measured quantities using one of the following methods (20):

11.4.1 For the non-multiplying example in accordance with 9.4.3.1:

$$m_{eff} = (R_c - a_0)/a_1 \quad (9)$$

11.4.2 From the calibration fit in accordance with 9.4.3.2 for count rates uncorrected for neutron multiplication, the corrected reals count rate is given by:

$$R_c = a_0 + a_1 m_{eff} + a_2 (m_{eff})^2 \quad (10)$$

Inverting this equation yields the  $^{240}\text{Pu}$  effective mass:

$$m_{eff} = \frac{-a_1 + \sqrt{a_1^2 - 4a_2(a_0 - R_c)}}{2a_2} \quad (11)$$

11.4.3 Other analysis procedures have been validated and documented. Details of these topics are beyond the scope of this test method. General information is included in Appendix X2 for reference.

11.5 The standard equation for calculating the  $^{240}\text{Pu}$  effective mass is a function containing nuclear constants for the even-mass plutonium isotopes and is specific to the coincidence circuitry (4, 21). The following equation from Reference 21 is one of the more commonly seen formulations of the  $m_{eff}$  equation.

$$m_{eff} = 2.52m_{238} + m_{240} + 1.68m_{242} \quad (12)$$

where:

$m_{xxx}$  = known mass of plutonium Isotope xxx in the material.

11.6 The total Pu in the scrap or waste package is determined by dividing  $m_{eff}$  by the effective  $^{240}\text{Pu}$  fraction assigned to the package.

$$\text{Pu mass} = m_{eff} / [2.52f_{238} + f_{240} + 1.68f_{242}] \quad (13)$$

where  $f_{xxx}$  denotes the weight fraction of isotope xxx.

11.7 Error estimates for the Pu mass should include all components which cause significant effects. These generally include: counting statistics, calibration errors (including reference material uncertainties), matrix uncertainties, item and nuclear material heterogeneities, and uncertainties in the isotopic ratios. Some components may be difficult to quantify. The random error standard deviation associated with the Pu mass and due solely to counting statistics can be derived from the measured counting statistics by standard error propagation methods (18). This value is usually computed and printed along with the Pu mass measurement.

## 12. Precision and Bias

NOTE 4—The precision and bias of passive neutron coincidence measurements are functions of several interrelated factors; consequently, a simple precision or bias statement is rarely possible. The interrelated factors include facility specific procedures, the quality of the scrap/waste segregation program, the appropriateness of the reference material matrix models, matrix types, chemical forms, and quantities. This section

provides information on the topic, but cannot substitute for critical thinking, professional skill, and verification measurements. The evaluation of the uncertainty for a passive neutron coincidence measurement is not a purely mathematical task; it requires detailed knowledge of the measurement method, the procedures, and the items being measured. Measurements of uncharacterized scrap and waste items can yield results of indeterminate bias. However, a combination of measurement methods applied to such items may be used to estimate the validity of the measurements. Except for measurements of small quantities, the possibility of bias is of greater concern than the issue of inadequate precision.

12.1 The precision of a passive neutron coincidence measurement can be estimated from replicate measurements. When passive neutron coincidence counters are set up and functioning properly, they follow a Poisson distribution (22). In cases where the Poisson assumption is valid, the precision may also be estimated using statistical calculations on data from a single measurement, such as that presented in Tables 1 and 2 (23).

12.1.1 The instrument calculated values of %  $\sigma_m$  ( $\sigma_m$  expressed as a percent) given in Table 3 are examples of the counting statistics precision that can be achieved with well-characterized material. Counting statistics contribute a random error of less than 1 % of the measured mass for 300-s measurements of items containing between 10g and 150g of high purity  $^{240}\text{PuO}_2$ . Table 1 indicates that, for pure and impure materials containing 0.6g of effective  $^{240}\text{Pu}$ ,  $\sigma_m$  ranges from 2.3 % to 11.7 % for 300-s measurements (23).

12.1.2 The repeatability and reproducibility of a passive neutron coincidence measurement can be estimated from replicate measurements. For a wide variety of measurements similar to those in Tables 3 and 1,  $\sigma_m$  approximately estimates the standard deviation that would be observed in a series of repeated measurements.

12.1.3 In general, longer counting times, larger quantities of nuclear material, and use of instruments with higher detection efficiencies will improve measurement precision.

12.1.4 Precision and bias are dependent upon many factors relating to the segregation and packaging of materials, as well as the physical and chemical form of the plutonium. For example, bias introduced by matrix differences can be minimized by a waste and scrap segregation plan and may be detected by monitoring changes in the ratio  $R/T$ . Also, if the characteristics of the material do not match the materials used in the calibration, the bias may increase indeterminately. Table 2 illustrates the percent relative difference between the measured and reference effective  $^{240}\text{Pu}$  masses for pure and slightly impure plutonium oxides.

**TABLE 1 Precision Data for Passive Neutron Coincidence Counter Measurements of Plutonium with Assorted Impurities**

NOTE 1—Measurement data from a set of 10 g plutonium disks (0.6 g of  $^{240}\text{Pu}$  effective) with assorted impurities (23). Measurement time was 300 s. %  $\sigma_m$  was calculated from the counting statistics of a single measurement and has been verified by replicate measurements.

| Item ID | Material                    | % $\sigma_m$ |
|---------|-----------------------------|--------------|
| A1-86   | $\text{PuO}_2$              | 3.0          |
| A1-87   | $\text{PuO}_2 + \text{A1}$  | 5.7          |
| A1-88   | $\text{PuO}_2 + \text{MgO}$ | 6.9          |
| A1-89   | $\text{PuO}_2 + \text{Si}$  | 3.4          |
| A1-90   | $\text{PuO}_2 + \text{B}$   | 11.7         |
| A1-91   | $\text{PuF}_4$              | 7.5          |
| A1-92   | Pu Metal                    | 2.3          |

**TABLE 2 Bias Resulting from Passive Neutron Coincidence Counter Measurements of Plutonium Oxide**

NOTE 1—Passive neutron coincidence counter measurement data for pure and slightly impure plutonium oxides (23). All measurement times were 300 s. Calibration parameters for these measurements were determined from pure  $\text{PuO}_2$  reference materials. The % Relative Difference is the difference between the measured and reference values expressed as a percent,  $\sigma_m$  is 0.8 %, or better, for these measurements.

| Item ID                       | Reference Total Pu Mass (g Pu) | Reference Effective $^{240}\text{Pu}$ Mass (g) | % Relative Difference |
|-------------------------------|--------------------------------|--|-----------------------|
| <i>Pure Plutonium Oxide</i>   |                                |  |                       |
| LAO261C10                     | 847.1                          | 144.4  | 0.42                  |
| LAO255C10                     | 542.9                          | 92.26  | −0.56                 |
| LAO252C10                     | 321.3                          | 54.37  | −2.40                 |
| <i>Impure Plutonium Oxide</i> |                                |  |                       |
| PEO381                        | 613.4                          | 65.02  | −3.71                 |
| PEO382D                       | 299.3                          | 29.70  | −3.81                 |

**TABLE 3 Precision Data for Passive Neutron Coincidence Counter Measurements of Well Characterized Material**

NOTE 1—Passive neutron coincidence counter precision data from a set of pure  $\text{PuO}_2$  items (23). The count time was 300 s for these measurements. %  $\sigma_m$ , determined from the counting statistics of a single measurement, was less than 1.0 % for each of the items.

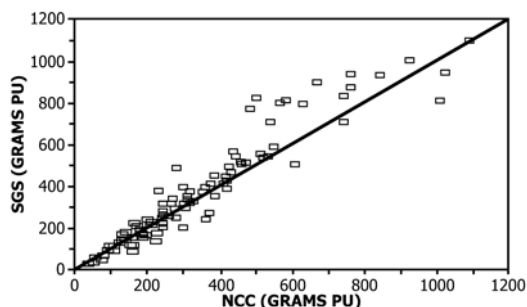
| Item ID   | Reference Total Pu Mass (g Pu) | Reference Effective $^{240}\text{Pu}$ Mass (g) | % $\sigma_m$ |
|-----------|--------------------------------|--|--------------|
| LAO250C10 | 59.87                          | 10.10  | 0.94         |
| LAO251C10 | 171.6                          | 29.29  | 0.82         |
| LAO252C10 | 321.3                          | 54.37  | 0.74         |
| LAO256C10 | 384.2                          | 65.15  | 0.72         |
| LAO255C10 | 542.9                          | 92.26  | 0.70         |
| LAO253C10 | 611.7                          | 104.6  | 0.6          |
| LAO261C10 | 847.1                          | 144.4  | 0.65         |
| LAO261C11 | 875.0                          | 149.2  | 0.64         |

12.2 Each user of this test method should determine the precision and bias for their specific scrap and waste categories (24).

12.2.1 In addition to the checks described in 9.3, a comparison of the results with another assay technique (such as segmented gamma-ray scanning, calorimetry, or destructive analysis) can be quite helpful. In general, two techniques based on different physical properties are susceptible to different sources of bias (25, 26).

12.2.2 Figs. 3 and 4 compare the results of passive neutron coincidence measurements with results derived from alternative techniques. Fig. 3 compares passive neutron measurements with segmented gamma scanner measurements (Test Method C1133) for 19 liter 5 gallon pails containing plutonium scrap (27). Fig. 4 compares passive neutron measurements with calorimetry (Test Method C1458) (28). These matrices do not contain large amounts of hydrogen and were generated at two different plutonium processing facilities. Both Figs. 3 and 4 suggest that an individual passive neutron coincidence counter measurement may be biased as much as 10 % to 20 % compared to the other method.

12.3 This technique measures the abundances of the even isotopes of plutonium. Biases in the determination of the relative abundances of the isotopes of plutonium will result in significant bias in the calculated total mass of plutonium. A



NOTE 1—The results of passive neutron coincidence measurements of the plutonium content of 5-US gal. pails are compared to the results of Segmented Gamma Scanner (SGS) measurements. The pails contain scrap and waste generated by a plutonium reprocessing facility in Aiken, South Carolina (26). The matrices have no hydrogen. The measurement uncertainties for both techniques are dominated by bias rather than precision.

**FIG. 3 Comparison of Neutron Coincidence Counter Measurements of Plutonium with SGS Measurements**

fractional bias in  $m_{eff}$  propagates to the same fractional bias in the total plutonium mass.

12.4 Reference materials are assigned plutonium mass and isotopic ratio values which have uncertainties associated with them. Calibrations are based on these “known” values. If there are biases in the “known” values of the reference materials, they will cause a bias in the neutron assay. Uncertainties in “known” values must be propagated into the calculated uncertainty of an assay.

12.5 Cosmic-ray background can be significant for small plutonium loadings in the presence of large quantities of high atomic number matrix. The bias effect is of the order of 0.02 g  $m_{eff}$  at sea level, and can double compared to sea level at an elevation of 2000 m (15).

12.6 If the detection efficiency is not constant over the assay volume, bias effects can occur due to item positioning or varying fill heights of the material in the container. The detection efficiency of some 208 1 systems has been determined to vary as much as 15 % for the totals and 28 % for coincidence count rates over the volume of the assay chamber (4, 8, 15, 25).

12.7 Neutron multiplication effects increase with plutonium mass, and are affected by geometrical variations in the distribution of the plutonium and the presence of moderating and ( $\alpha$ , n) producing materials.

12.7.1 The nonlinearity of the uncorrected  $\text{PuO}_2$  calibration curve in Fig. 2 is attributable to neutron multiplication.

12.7.2 Neutrons from ( $\alpha$ , n) reactions in low atomic number matrices can induce fissions also. This will bias the result high unless the multiplication correction technique is used. (See Appendix X2.)

12.7.3 Multiplication effects are larger for counting chambers without a cadmium liner.

12.8 The hydrogen content (water, plastic, acid, etc.) of an assay item may increase the detection efficiency and multiplication effects by lowering the average neutron energy, thereby causing a bias. The largest potential inaccuracies associated with nonuniform source distributions in passive neutron coincidence counters is found when large amounts of moderating material are contained in the scrap or waste matrix. Average to minimum passive response ratios of 4.5 have been reported for highly moderating matrices with hydrogen densities above 0.04 g  $m_{eff}$  (29). In severe cases where the hydrogen content varies unexpectedly from the reference materials used for calibration, the effect can cause the plutonium mass result to double for 50 g  $m_{eff}$  (30).

12.9 Item container wall effects, for example, polyethylene liners, have biased individual assay results as much as 7 %. It is important to standardize waste containers, preferably using materials which do not absorb or moderate neutrons.

12.10 Measurements of plutonium items with uranium contamination will be biased unless corrections are made (31).

12.11 The presence of modest amounts of neutron absorbers in the matrix does not cause a bias in passive neutron coincidence measurements because only thermal neutrons are absorbed by neutron absorbing materials and a change in the number of thermal neutrons will not affect the coincidence count rate because these neutrons 1) move too slowly to appear in coincidence or, 2) do not pass through the cadmium liner or polyethylene present in most passive neutron coincidence counters.

12.12 Mixing other spontaneous fission isotopes with the Pu will increase  $R$ . These materials must be segregated and assayed by another method, unless a correction is made in the  $m_{eff}$  formula. Curium has been a problem in high burn-up Pu.

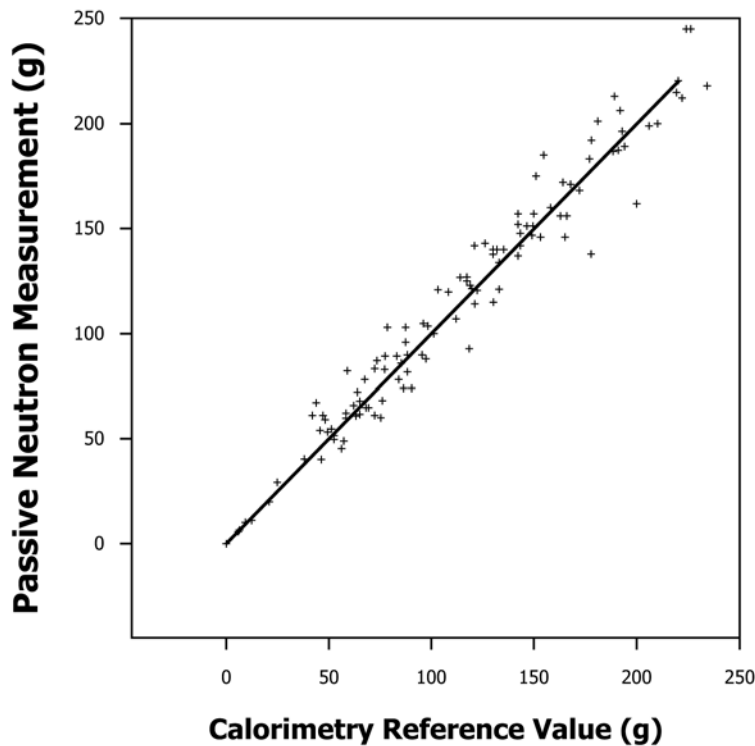
12.13 The density of the Pu compound can have significant effects on  $R$ . An increase in the Pu density increases the item multiplication.  $R$  can vary as much as 10 % for a can of oxide if the can is tumbled several times to fluff up the powder (28).

12.14 The instability of modern electrical circuits contributes a negligible error (<0.1 %) to the results (32). Proper adjustment of parameters such as the pre-delay and the gate length cause their potential for bias effects to become negligible.

### 13. Keywords

13.1 nondestructive assay; passive neutron coincidence counting; plutonium; scrap and waste





NOTE 1—The results of passive measurements of the plutonium content of small containers of scrap and waste are compared to the results of calorimetry and gamma-ray isotopic measurements (28). The matrices do not contain hydrogen. The measurement uncertainties are dominated by bias rather than precision.

FIG. 4 Comparison of Neutron Coincidence Counter Measurements of Plutonium with Results Obtained from Calorimetry and Gamma-Ray Isotopics Analysis

## APPENDIXES

### (Nonmandatory Information)

#### X1. USE OF THE $R/T$ RATIO FOR EVALUATING ASSAY RESULTS

X1.1 Some applications have used the Reals-to-Totals ratio,  $R/T$ , to help evaluate the suitability of the selected calibration curve (25). The following information is presented here to help the potential user.

X1.1.1 For the particular case of the high-level neutron coincidence counter, (HLNCC-II), non-multiplying plutonium metal has an  $R/T$  ratio of approximately 0.1. For  $^{252}\text{Cf}$  the  $R/T$  ratio is approximately 0.18; for non-multiplying plutonium oxide, the  $R/T$  ratio ranges from 0.04 to 0.08. With increasing  $(\alpha, n)$  contribution, these ratios get much smaller.

X1.1.2 As the  $R/T$  ratio approaches zero, it indicates that fewer of the neutrons are from fission events. In this case, the induced fission rate in  $^{239}\text{Pu}$  may become a significant portion of the total fission rate. The usual consequence of this is that the assay result is biased high. The selected calibration curve may not be suitable and alternative analysis methods are needed (25).

## X2. METHODS FOR TREATING NEUTRON MULTIPLICATION OF WELL-CHARACTERIZED MATERIAL

X2.1 The following information is included to show methods which have been applied to well-characterized material in which there is known neutron multiplication or for the case where there is little neutron count rate.

X2.1.1 *Known Multiplication Method (3)*—For similar geometries and plutonium loadings the neutron multiplication  $M$  is assumed to be related to the  $^{239}\text{Pu}$  mass.  $M$  is obtained from the reference materials or Monte Carlo calculations, while the measured quantities  $R_c$  and  $T_c$  are used to solve for the  $(\alpha, n)$  component and  $m_{eff}$ .

X2.1.2 *Multiplication Corrected Reals  $R_{MC}$  (3)*—Compute the  $(\alpha, n)$  effects from the known chemical composition. Compute  $M$ , then use  $R_c$ , and  $M$  to compute  $R_{MC}$ . A linear

relationship should exist between  $R_{MC}$  and  $m_{eff}$ . When the chemical composition of the plutonium is known, this approach gives the most accurate results for the widest range of material categories with a single calibration.

X2.1.3 *Self-Interrogation Method (24)*—This technique requires the induced fission response to be comparable to or larger than the spontaneous fission response.

X2.1.4 For very small plutonium loadings, a more sensitive upper limit determination of the amount of Pu in the item may be achieved from relating  $m_{eff}$  to the Totals count rate. The Totals count rate is however more susceptible to background and matrix effects than is the reals count rate  $R$ .

## X3. COMPARISON OF THE (R+A) AND T REGISTERS

X3.1 A feature of the shift register coincidence circuit is that the  $R+A$  coincidence sum can exceed the totals  $T$  during a measurement due to the combinatorial action of the shift

register. When the  $R+A$  sum exceeds the Totals sum, the operator should not assume that the shift register has malfunctioned or that the data are invalid.

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