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Standard Test Methods for NONDESTRUCTIVE ASSAY OF SPECIAL NUCLEAR MATERIALS CONTAINED IN SCRAP AND WASTE[1]

This standard is issued under the fixed designation C 853; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These methods cover procedures for the nondestructive assay of special nuclear material (SNM) contained in scrap and waste.

1.2 Guidelines regarding the assay of SNM, for example, the choice of a method, analysis prerequisites, interference, etc., are presented in U.S. Atomic Energy Commission Regulatory Guide (**1, 2**).

1.3 The procedures appear in the following order:

	Sections
Nondestructive Determination of Plutonium in Containers of 4 Litres or Less or Fabrication Waste By Neutron-Coincidence Counting	3 to 11
Nondestructive Determination of Plutonium In Containers of 1 Litre or Less in Fabrication Scrap By Neutron-Coincidence Counting	12 to 19
Nondestructive Determination of Plutonium in 55 and 30-Gallon Drums of Combustible Fabrication Wastes By Neutron Coincidence Counting	20 to 28
Nondestructive Analysis of Plutonium in Fabrication Scrap and Waste in Containers of 20 Litres or Less By Segmented Passive Gamma-Ray Scanning	29 to 36
Nondestructive Determination of Transuranic Radioisotope Activity in Low Density Combustible Waste at the 10 nCi/g Level	37 to 44

NOTE 1—Additional procedures, involving various analysis techniques, are under study for incorporation in this standard at a later date.

2. Safety Precautions

NOTE 2—A discussion of safety procedures is not within the scope of this standard. Competent health and nuclear safety personnel should be consulted in regard to the precautions listed.

2.1 Proper precautions should be taken to prevent inhalation or ingestion of uranium or plutonium during handling operations.

2.2 Proper precautions should be taken to prevent overexposure to radiation from SNM during handling operations.

2.3 Proper precautions should be taken regarding criticality control during handling and storage of both waste samples and standards.

2.4 Standard packages should be surveyed on a regular basis with an alpha monitor to verify the contained integrity of the packages.

NONDESTRUCTIVE DETERMINATION OF PLUTONIUM IN CONTAINERS OF 4 LITRES OR LESS OF FABRICATION WASTE BY NEUTRON-COINCIDENCE COUNTING

3. Scope

3.1 This method covers the nondestructive determination of plutonium in noncombustible- and combustible-type waste contained in packages of a volume of 4 litres or less by neutron-coincidence counting. The applicable concentration range of the method is from 0.01 to 100 g effective ^{240}Pu mass.

NOTE 3—The effective ^{240}Pu mass ($^{240}Pu_{eff}$) is defined in 7.4.

3.2 The coincident neutron-counting method is necessary for accurate assays due to high (α, n) yield interferences from low Z nuclei associated with the plutonium (for example, O, C, F, Be, etc.). Typically gross-neutron counting is only applicable for very well-characterized

[1] These methods are under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle.

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samples, a case generally not true for plutonium-bearing combustible wastes. Because the assay is based on the specific spontaneous fission rates of the various plutonium isotopes, the isotopic composition of the plutonium must be known prior to the assay. The plutonium isotopic composition is obtained by mass spectrometry, gamma-ray analysis, or from the operating history of the waste producer.

3.3 This method may be applied to high- and low-density materials in packages in irregular shape in a standard container that will easily fit into the counting well.

4. Summary of Method

4.1 The method involves the detection of coincident neutrons from the spontaneous fission of ^{238}Pu, ^{240}Pu, and ^{242}Pu. The detectors are BF_3– or ^3He–filled proportional counters that are imbedded in polyethylene and surround the sample to be assayed. The container for analysis is placed in the counting well. The analysis is initiated and neutron events are registered in scalers. Gross neutrons, coincident neutrons plus accidental events, and accidental events are recorded or determined. The accidental events are subtracted from the coincident plus accidental events, an amplifier dead-time correction is made, if appropriate, and the net coincident events are related to effective ^{240}Pu mass by a calibration curve. The net coincident events are a relative measure of the spontaneous fissions from ^{238}Pu, ^{240}Pu, and ^{242}Pu. Accurate analyses are dependent on the use of isotopically equivalent standards or prior knowledge of the isotopic distribution of the plutonium in the sample.

4.2 A detailed description of neutron-well coincidence counters and their characteristics is available **(3, 4)**.

5. Interferences

5.1 Neutron-multiplication effects may become severe at high-Pu sample loadings **(5)**. Typical techniques used to control this interference are: (*1*) equivalent reference standards used for calibration, or (*2*) use of source addition techniques.

5.2 Packages containing large quantities (>0.1 % fluoride equivalent) of low Z elements, for example, F, Be, B, etc., must be treated by other procedures capable of handling such samples. This condition is indicated by an unusually high-gross-neutron count and an unusually low-real-coincidence count.

6. Apparatus

6.1 *Neutron-Coincidence-Well Counter System*—Suitable components or systems, or both, are commercially available **(3, 6)**. The system should have the following minimum capability:

6.1.1 A cadmium-lined moderated counting well with BF_3 or ^3He detector tubes,

6.1.2 Discriminator circuitry,

6.1.3 Scalers for data readout,

6.1.4 Elapsed time readout,

6.1.5 Coincidence-logic unit capable of handling high-counting rates (Note 4), and

6.1.6 Partial cadmium sleeves should be used according to manufacturer's instructions, to reduce the neutron-count rate at high-Pu loadings.

NOTE 4—Shift register electronics **(7)** provide this capability.

6.2 *High Voltage Power Supply*—0 to 3000 V d-c is generally satisfactory.

6.3 *Amplifier,* commercially available low-noise amplifier with long-term gain stability of better than 0.05 %.

6.4 *Shielding*, 152.4 mm of a moderator, for example, paraffin or polyethylene surrounding the counting well. This well should be provided with an external cadmium shield of approximately 0.76 mm. Such shielding is optional if the ambient-neutron activity is low and constant.

7. Preparation of Standards

7.1 Standards are prepared by randomly distributing packages containing 0.01 to 1.0 g $^{240}Pu_{eff}$ throughout a container filled with matrix material characteristic of the type of material in the waste category being analyzed. A series of these synthetic standards are fabricated covering the ^{240}Pu mass range of interest for the preparation of a calibration curve. Standards should conform as much as possible to the sample with respect to the container size and the contents, including the matrix, SNM chemical form, and distribution.

7.2 Weigh accurately, to ± 0.005 g or better, plutonium-standard material, of known Pu and isotopic content, and traceable to an accepted standard reference material, into a suitable vial (metal or plastic) and seal.

7.3 Seal the vial in plastic.

7.4 Mark each bag with the material identification and the effective ^{240}Pu mass.

The effective ^{240}Pu mass is defined by the following equation:

$$240_{m_{eff}} = 240_m + \left(\frac{242_n}{240_n}\right) 242_m + \left(\frac{238_n}{240_n}\right) 238_m,$$

where:

m = the actual mass of the isotope in the package, and

n = the number of neutrons per gram of isotope per second emitted in the spontaneous fission of isotope.

The value for $^{242}n/^{240}n$ is 1.62 and for $^{238}n/^{240}n$ is 2.60.

7.5 Prepare sufficient packages to cover the mass range of interest.

8. Calibration

8.1 Obtain a minimum of four calibration points over the mass range of interest using standards that are representative of the materials being measured.

8.2 Independently calibrate the NDA system(s) for each category of scrap or waste to be assayed.

8.3 Within each category, measure the variation due to interference effects within the boundaries defining the limits of that category. For calibration standards, employ containers identical to those to be employed for the scrap or waste. Mock up their contents to represent the range of matrix conditions to be encountered.

8.4 Do not extrapolate beyond the calibration range.

8.5 Standard packages are randomly distributed in a matrix and container similar to the sample container and carried through the analysis procedures.

9. Procedure

9.1 *Instrumental Parameters:*

9.1.1 *High Voltage (typically 2000 to 3000 V)*—Determine the voltage setting from high-voltage plateau curves taken when the equipment is initially calibrated.

9.1.2 *Amplifier Gain*—Adjust according to manufacturer's instructions.

9.1.3 *Rise Time*—Adjust according to manufacturer's instructions.

9.1.4 *Discrimination (approximately 0.2 to 0.5 V)*—Determine the specific setting from a discriminator-bias curve taken when the equipment is calibrated.

9.1.5 *Counting Time*—The counting time should be sufficient to obtain desired counting statistics, including the background contribution. If $N = R - A$, then use the following equation to calculate the standard deviation:

$$\sigma_N = (\sigma_R^2 + \sigma_A^2)^{1/2}$$

9.2 *Analysis:*

9.2.1 Place the sample in the counting well, reset all registers, and initiate the analysis.

9.2.2 Set counting to terminate automatically when the preset time has elapsed.

9.2.3 Record the number of gross neutrons, G, the number of coincident neutrons plus accidental events, R, the number of accidental events, A, and the elapsed count time, T_ℓ.

10. Calculations

10.1 The number of net coincident events, N, is determined by the following equation:

$$N = \frac{N_o'}{\left(1 - \frac{DG_o'}{T_\ell}\right)^3},$$

where:

N_o' = observed net coincident events registered during analysis in the live-counting time mode, or $R - A$,

D = amplifier dead time given, μs. This constant is supplied by the equipment manufacturer and should be checked on a regular basis,

G_o' = number of gross neutron events observed while counting in the live-time mode, and

T_ℓ = total elapsed time, μs, while counting in the live-time mode, $T_\ell \geq$ the nominal time count when operating in the live-time mode.

An equivalent alternative in accordance with the manufacturer's instructions for the specific coincidence unit used is applicable.

10.2 The $^{240}Pu_{eff}$ is determined from a calibration curve of $^{240}Pu_{eff}$ versus N.

10.3 The total plutonium in the waste package is determined by dividing the $^{240}Pu_{eff}$ in grams by the effective plutonium −240 fraction assigned to the package, that is defined as the $^{240}Pu_{eff}$ mass divided by the total plutonium mass in the standard.