Designation: C1030 – 10 (Reapproved 2018)

Standard Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry[1]

This standard is issued under the fixed designation C1030; 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 This test method is applicable to the determination of isotopic abundances in isotopically homogeneous plutonium-bearing materials. This test method may be applicable to other plutonium-bearing materials, some of which may require modifications to the described test method.

1.2 The procedure is applicable to items containing plutonium masses ranging from a few tens of milligrams up to the maximum plutonium mass allowed by criticality limits.

1.3 Measurable gamma ray emissions from plutonium cover the energy range from approximately 30 keV to above 800 keV. K-X-ray emissions from plutonium and its daughters are found in the region around 100 keV. This test method has been applied to all portions of this broad spectrum of emissions.

1.4 The isotopic abundance of the ^{242}Pu isotope is not directly determined because it has no useful gamma-ray signature. Isotopic correlation techniques may be used to estimate its relative abundance Refs **(1)** and **(2)**.[2]

1.5 This test method has been demonstrated in routine use for isotopic abundances ranging from 99 to <50 % ^{239}Pu. This test method has also been employed for isotopic abundances outside this range.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets

C698 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Mixed Oxides ($(U, Pu)O_2$)

C982 Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems (Withdrawn 2008)[4]

C1207 Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting

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

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

C1493 Test Method for Non-Destructive Assay of Nuclear Material in Waste by Passive and Active Neutron Counting Using a Differential Die-Away System (Withdrawn 2018)[4]

C1500 Test Method for Nondestructive Assay of Plutonium by Passive Neutron Multiplicity Counting

E181 Test Methods for Detector Calibration and Analysis of Radionuclides

E267 Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances

[1] This test method 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.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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

[4] The last approved version of this historical standard is referenced on www.astm.org.

2.2 *ANSI Standards:*[5]

ANSI/IEEE Std 325-1996 IEEE Standard Test Procedures for Germanium Gamma-Ray Detectors

ANSI N15.36 Measurement Control Program – Nondestructive Assay Measurement Control and Assurance

3. Summary of Test Method

3.1 The intensities of gamma-rays emitted from a plutonium-bearing item are determined from a gamma-ray spectrum obtained with a High-Purity Germanium (HPGe) detector. The method has also been used with CdTe detectors.

3.2 The atom ratio, N^i/N^k, for isotopes i and k is related to the photopeak counting intensity, $C\,(E_j^i)$, for gamma ray j with energy E_j emitted from isotope i by:

$$\frac{N^i}{N^k} = \frac{C\,(E_j^i)}{C\,(E_l^k)} \cdot \frac{T_{1/2}^i}{T_{1/2}^k} \cdot \frac{BR_l^k}{BR_j^i} \cdot \frac{RE(E_l)}{RE(E_j)} \tag{1}$$

where:

$RE(E_i)$ = relative detection efficiency for a gamma-ray of energy E_i,

$T_{1/2}^i$ = half-life of isotope i, and

BR_j^i = gamma-ray branching ratio or branching intensity (usually expressed as gamma-rays per disintegration) of gamma ray j from isotope i.

3.3 The half lives $T_{1/2}$ and the branching ratios BR are known, published nuclear data. The photopeak counting intensity $C(E)$ is determined from the gamma ray spectrum of the measured item.

3.4 The relative detection efficiency, $RE(E)$, is a function of gamma-ray energy and arises from the combined effects of detector response, attenuation due to absorbers and container walls, and self-absorption within the measured item for gamma-rays of differing energies. The relative detection efficiencies are determined for each measured item from the observed gamma spectrum by considering a series of gamma rays from a single isotope. The quotient of the photopeak counting intensity for gamma ray j with energy E_j emitted from isotope i and the branching ratio of gamma ray j from isotope i is proportional to the relative detection efficiency at energy E_j. This quotient defines the shape of the relative efficiency as a function of energy.

$$\frac{C(E_j^i)}{BR_j^i} \alpha \left(\frac{N^i}{T_{1/2}^i} \right) \cdot RE(E_j) \tag{2}$$

3.5 All factors in Eq 1 are either determined from the gamma ray spectrum of the measured item or are known, published nuclear constants. The absolute atom ratios are determined without recourse to standards or calibration by this so-called Intrinsic Calibration technique.

4. Significance and Use

4.1 The determination of plutonium isotopic composition by gamma-ray spectrometry is a nondestructive technique and when used with other nondestructive techniques, such as calorimetry (Test Method C1458) or neutron counting (Test Methods C1207, C1316, C1493, and C1500), can provide a wholly nondestructive plutonium assay necessary for material accountancy and safeguards needs.

4.2 Because gamma-ray spectrometry systems are typically automated, the routine use of the test method is fast, reliable, and is not labor intensive. The test method is nondestructive, requires no sample preparation, and does not create waste disposal problems.

4.3 This test method assumes that all plutonium in the measured item has the same isotopic distribution, often called isotopic homogeneity (see 7.2.4 and 7.2.5).

4.4 The ^{242}Pu abundance is not measured by this test method and must be estimated from isotopic correlation techniques, stream averages, historical information, or other measurement techniques.

4.5 Americium-241 is a daughter product of ^{241}Pu. The ^{241}Am/^{239}Pu atom ratio can also be determined by means of this test method (assuming a homogeneous isotopic distribution of plutonium and ^{241}Am). The determination of the ^{241}Am/^{239}Pu atom ratio is necessary for the correct interpretation of a calorimetric heat measurement.

4.6 The isotopic composition of a given batch or item of plutonium is an attribute of that item and, once determined, can be used in subsequent inventory measurements to verify the identity of an item within the measurement uncertainties.

4.7 The method can also measure the ratio of other gamma-emitting isotopes to plutonium assuming they have the same spatial distribution as the plutonium in the item. Some of these "other" gamma-emitting isotopes include isotopes of uranium, neptunium, curium, cesium, and other fission products. The same methods of this standard can be used to measure the isotopic composition of uranium in items containing only uranium (**3**, **4**, **5**, **6**).

5. Interferences

5.1 Because of the finite resolution of even the best quality HPGe detectors, the presence of other gamma-emitting sources must be assessed for their effects on the isotopic abundance determination.

5.1.1 The detector used for the spectral measurements shall be adequately shielded from other nearby plutonium sources. Background spectra shall be collected to ensure the effectiveness of detector shielding and to identify the background radiations.

5.1.2 If fission products are present in the item being measured, they will contribute additional gamma-ray spectral peaks. These peaks occur mainly in the 500 to 800-keV energy range and may affect the intensity determination of plutonium and americium peaks in this region. These high-energy gamma-rays from fission products also produce contributions to the Compton background below 500 keV that decrease the precision for peak intensity determination in this region.

5.1.3 For mixed plutonium-uranium oxide-bearing items, the appropriate corrections for the spectral peaks produced by uranium gamma emission shall be applied. The main interferences from uranium are listed in Table 1.

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