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Designation: ~~C1268 – 15~~ C1268 – 23

Standard Test Method for Quantitative Determination of ^{241}Am in Plutonium by Gamma-Ray Spectrometry[1]

This standard is issued under the fixed designation C1268; 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 covers the quantitative determination of ^{241}Am by gamma-ray spectrometry in plutonium nitrate solution samples that do not contain significant amounts of radioactive fission products or other high specific activity gamma-ray emitters.

1.2 This test method can be used to determine the ^{241}Am in samples of plutonium metal, oxide and other solid forms, when the solid is appropriately sampled and dissolved.

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1.3 The values stated in SI units are to be regarded as standard. Additionally, the non-SI units of electron volts, kiloelectron volts, and liters are to be regarded as standard. No other units of measurement are included in this standard.

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

1.5 *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.*

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

C758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal
C759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions
C859 Terminology Relating to Nuclear Materials
C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry
C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis
C1592/C1592M Guide for Making Quality Nondestructive Assay Measurements
~~E181~~E3376 ~~Guide~~Practice for ~~Detector~~ Calibration and ~~Analysis of Radionuclides~~ Usage of Germanium Detectors in Radiation Metrology for Reactor Dosimetry

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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[2] 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.

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2.2 *U.S. Nuclear Regulatory Commission Regulatory Guides:*[3]

Regulatory Guide 5.9, Rev. 2—Guidelines for Germanium Spectroscopy Systems for Measurement of Special Nuclear ~~Materials~~Material

Regulatory Guide 5.53, Rev. 1—Qualification, Calibration, and Error Estimation Methods for Nondestructive Assay[3]

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

4. Summary of Test Method

4.1 An aliquot of the sample that contains about ~~10~~10 ng to 100 ng of ^{241}Am is analyzed by measuring the intensity of the characteristic 59.5 keV gamma ray emitted by ^{241}Am.

4.2 Multiple sample geometries may be used if an appropriate calibration for each geometry is made.

4.3 The sample geometry must be reproducible. This includes the physical characteristics of the sample container, the positioning of the sample, and the volume of sample viewed by the gamma-ray detector.

4.4 Electronic corrections are made, if required, for the effects of pulse pile-up and dead time losses due to the activity of the sample. The necessity of dead time and pulse pile-up corrections can be reduced by sample dilution to control count rates.

4.5 A correction is made for the contribution to the 59.5 keV intensity due to gamma rays produced in the decay of ^{237}U.

4.6 The relationship between the measured gamma-ray intensity and the ^{241}Am content is determined by the use of appropriate standards.

5. Significance and Use

5.1 This test method allows the determination of ^{241}Am in a plutonium solution without separation of the americium from the plutonium. It is generally applicable to any solution containing ^{241}Am.

5.2 The ^{241}Am in solid plutonium materials may be determined when these materials are dissolved (see Practice C1168).

5.3 When the plutonium solution contains unacceptable levels of fission products or other materials, this method may be used following a tri-n-octylphosphine oxide (TOPO) extraction, ion exchange or other similar separation techniques (see Test Methods C758 and C759).

5.4 This test method is less subject to interferences from plutonium than alpha counting since the energy of the gamma ray used for the analysis is better resolved from other gamma rays than the alpha particle energies used for alpha counting.

5.5 The minimal sample preparation reduces the amount of sample handling and exposure to the analyst.

5.6 This test method is applicable only to homogeneous solutions. This test method is not suitable for solutions containing solids.

5.7 Solutions containing ^{241}Am at concentrations as little as 1×10^{-5} g/L ~~^{241}Am~~ may be analyzed using this method. The lower limit depends on the detector used and the counting geometry. Solutions containing high concentrations may be analyzed following an appropriate dilution.

6. Interferences

6.1 The presence of other radioactive nuclides in the sample or in the vicinity of the detector may produce interferences. These

[3] Available from U.S. Nuclear Regulatory Commission, One White Flint North, 11555 Rockville Pike, Rockville, MD 20852. Also through www.nrc.gov.

may be due to the Compton scattering of high energy gamma rays which contribute to the background in the region of interest or from gamma rays with energies close to the energies used for the analysis.

6.2 The presence of ^{237}U will interfere if a correction is not applied. This interference will lead to an over estimation of the amount of ^{241}Am present. This interference is especially pronounced in plutonium from which the americium has recently been separated.

6.3 The presence of radioactive materials in the vicinity of the gamma-ray detector which are not in the sample may create interferences if detector shielding is not adequate. These interferences may be due to the Compton scattering of high energy gamma rays which contribute to the background in the region of interest or from gamma rays with energies close to the energies used for the analysis.

7. Apparatus

7.1 *High-Resolution Gamma Ray Counting System*—A high resolution gamma-ray counting system is required. General guidelines for the selection of detectors and signal processing electronics are discussed in NRC Regulatory Guide 5.9. Data acquisition systems are addressed in NRC Regulatory Guide 5.9. This system should include the following items as a minimum.

7.1.1 *Germanium Photon Detector with Integral Preamplifier*—A coaxial type detector should typically have a full width at half maximum resolution of 850 eV or less at ~~122 keV~~ 122 keV and 2.0 keV or less at 1332 keV. A planar type detector should typically have a full width at half maximum resolution of 600 eV or less at 122 keV. Consideration should be given to the use of a high efficiency detector to enhance the ability to analyze low levels of americium.

7.1.2 *High Voltage Power Supply*—A high voltage power supply with voltage range and current output compatible with the detector selected is required. It is desirable that the voltage output be continuously adjustable.

7.1.3 *Nuclear Spectroscopy Amplifier*—Select a nuclear spectroscopy amplifier with pulse shaping, baseline restoration, and pulse pile-up rejection circuitry.

7.1.4 *Multichannel Pulse Height Analyzer (MCA)*—Select an MCA with a minimum of 2048 channels. It is desirable that the MCA be compatible with computerized operations so that data acquisition and analysis may be automated. The analog to digital converter (ADC) associated with the MCA should have a clock rate of at least 100 MHz and the capability of digitizing the input voltage range into a minimum of 2048 channels (other types of ADC's which provide equivalent capabilities can be used). The ADC should also have dead time and pulse pile-up correction capabilities.

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7.2 *Sample Holder,* incorporating shielding to limit the interferences from background radiation sources, is required. Collimation to restrict the view of the detector to a portion of the sample may be required. The sample holder may incorporate more than one sample position. The sample holder shall provide reproducible positioning for each sample position so that a consistent volume or portion of the sample is viewed by the detector.

7.3 *Sample Vials* of sufficient volume to contain the desired sample as described in 10.2 are required. The sample vials should be made of low density materials and have reproducible dimensions such as wall thickness and internal diameter. Vials with identical dimensions should be used for samples and standards.

8. Hazards

8.1 ~~Plutonium and americium bearing materials are radioactive and toxic. Adequate laboratory facilities, gloveboxes, fume hoods, and so forth, along with safe techniques must be used in handling samples containing these materials. A detailed discussion of all the precautions necessary is beyond the scope of this test method; however, personnel who handle these materials should be familiar with such safe handling practices.~~This standard involves work with nuclear materials. The unique hazards and controls required to conduct the work contained in this standard from a safety, environmental, and security standpoint is the responsibility of the facility operators, in compliance with any applicable regulations. Any information given for handling these types of materials contained herein is meant only as a guide for consideration of the user and not a requirement.

8.2 Solutions and solids containing radioactive materials represent a potential for high radiation exposure to personnel handling them. Appropriate sample shielding, sample handling procedures, and radiation monitoring should be employed to ensure personnel protection.

9. Calibration and Standardization

9.1 Calibrate the counting system for energy (eV/channel) in the range ~~0~~0 keV to 300 keV using a radioactive source or sources which emit gamma rays with well known energies. A plutonium source is an obvious choice. See ~~Methods~~Practice ~~E181~~E3376, Guide C1592/C1592M, and U.S. NRC Regulatory Guide 5.53 for further guidance.

9.2 Determine the relative detection efficiency (counts/emitted gamma ray) of the counting system in the ~~0 to 300 keV~~ 0 keV to 300 keV range. Specifically, the efficiency at ~~59.5 keV and 208 keV~~ 59.5 keV and 208 keV needs to be determined. See ~~Methods~~Practice ~~E181~~E3376, Guide C1592/C1592M and U.S. NRC Regulatory Guide 5.53 for further guidance.

9.3 The relationship between the mass of ^{241}Am and the number of ~~59.5 keV~~ 59.5 keV gamma rays is established through fundamental physics and basic nuclear constants, that is, the number of ~~59.5 keV gamma rays/sec/gram~~ 59.5 keV gamma rays per second per gram of ^{241}~~Am = 4.543 × 10~~Am equals 4.543 × 10^{10}.

10. Procedure

10.1 If necessary, prepare a plutonium solution from a solid sample following the procedure in Practice C1168 or other dissolution procedure.

10.2 Determine the amount of solution and the dilution required to provide ~~10~~10 ng to 100 ng of ^{241}Am in the selected sample volume. The sample volume viewed by the detector should be consistent for the samples and standards used, regardless of the concentration.

10.3 Determine the counting time necessary to achieve the desired statistical counting precision. Samples which contain more americium will generally require less time to achieve the same statistical precision.

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10.4 Quantitatively transfer the predetermined volume of solution from ~~9.2~~10.2 into a sample vial and close.

10.5 Place the vial in the counting system sample holder and acquire a spectrum. The detector should see a consistent portion of the sample volume. The same counting geometry and sample size as used for the standards must be used.

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10.6 Record the sample counting time, sample volume, dilution factor, and counting geometry used if more than one is available.

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11. Calculation

11.1 Using the same methods as used for the calibration, determine the background corrected net count rates for the ~~59.5 keV~~ 59.5 keV gamma ray and the 208 keV gamma ray using the spectral data acquired in 10.5.

11.2 Calculate the 59.5 keV counting rate due to ^{241}Am in the sample.

$$R_{\mathrm{Am}}(59) = \frac{R_{\mathrm{obs}}(59)/D(59) - B_{\mathrm{U}} R_{obs}(208)/D(208)}{1 - B_{\mathrm{U}}/B_{\mathrm{Am}}} \tag{1}$$

where:

~~$R_{\mathrm{Am}}(59)$~~	=	~~59.5 keV rate (gamma rays/s) due to ^{241}Am,~~
$R_{\mathrm{Am}}(59)$	=	59.5 keV rate (gamma rays per second) due to ^{241}Am,
~~$R_{obs}(59)$~~	=	~~measured 59.5 keV rate (counts/s),~~
$R_{obs}(59)$	=	measured 59.5 keV rate (counts per second),
~~$D(59)$~~	=	~~detection efficiency (counts/gamma ray) at 59.5 keV,~~
$D(59)$	=	detection efficiency (counts per gamma ray) at 59.5 keV,
~~$R_{obs}(208)$~~	=	~~measured 208 keV rate (counts/s),~~
$R_{obs}(208)$	=	measured 208 keV rate (counts per second),
~~$D(208)$~~	=	~~detection efficiency (counts/gamma ray) at 208 keV,~~
$D(208)$	=	detection efficiency (counts per gamma ray) at 208 keV,
B_{U}	=	1.5668, and
B_{Am}	=	45385.6.