**Designation:** ~~C1268 – 94 (Reapproved 2008)~~ C1268 – 15

# Standard Test Method for
# Quantitative Determination of ²⁴¹~~Americium 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~~americium 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~~americium 241~~ Am in samples of plutonium metal, oxide and other solid forms, when the solid is appropriately sampled and dissolved.

1.3 The values stated in SI units 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 practices and determine the applicability of regulatory limitations prior to use.*

## 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

~~C982 Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems (Withdrawn 2008)[3]~~

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 Test Methods for Detector Calibration and Analysis of Radionuclides

~~2.2 *ANSI Standards:*[4]~~

~~ANSI N15.20 Guide to Calibrating Nondestructive Assay Systems~~

~~ANSI N15.35 Guide to Preparing Calibration Material for Nondestructive Assay Systems that Count Passive Gamma Rays[4]~~

~~ANSI N15.37 Guide to the Automation of Nondestructive Assay Systems for Nuclear Material Control[4]~~

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

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.

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[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. Current edition approved ~~Jan. 1, 2008~~June 1, 2015. Published ~~February 2008~~July 2015. Originally approved in 1994. Last previous edition approved in ~~2000~~2008 as C1268 – 94 ~~(2000).~~(2008). DOI: ~~10.1520/C1268-94R08.~~10.1520/C1268-15.

[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.

[3] Available from U.S. Nuclear Regulatory Commission, ~~1717 H ST., NW, Washington, DC 20555.~~One White Flint North, 11555 Rockville Pike, Rockville, MD 20852. Also through www.nrc.gov.

## 4. Summary of Test Method

4.1 An aliquot of the sample that contains about 10 to 100 ng of $^{241}$americium 241 Am is analyzed by measuring the intensity of the characteristic 59.5 keV gamma ray emitted by $^{241}$americium 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 $^{237}$uranium 237. U.

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

## 5. Significance and Use

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

5.2 The $^{241}$americium 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 as little as $1 \times 10^{-5}$ g/L $^{241}$americium 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 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 $^{237}$uranium 237 U will interfere if a correction is not applied. This interference will lead to an over estimation of the amount of $^{241}$americium 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 Guide C982 and NRC Regulatory Guide 5.9. Data acquisition systems are addressed in ANSI 15.37 and 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 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.

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 9.210.2 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, gloved boxes, gloveboxes, fume hoods, etc.,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.

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 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 E181, ANSIGuide C1592/C1592MN15.20,, and U.S. 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 range. Specifically, the efficiency at 59.5 keV and 208 keV needs to be determined. See Methods E181, ANSI N15.20Guide C1592/C1592M and U.S. Regulatory Guide 5.53 for further guidance.

9.3 The relationship between the mass of $^{241}$americium 241 Am and the number of 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 $^{241}$americium 241 = 4.543 × 10Am = 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 to 100 ng of $^{241}$americium 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.

10.4 Quantitatively transfer the predetermined volume of solution from 9.29.2 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.

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

## 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 gamma ray and the 208 keV gamma ray using the spectral data acquired in 9.510.5.

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

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