



Standard Test Method for Plutonium Assay by Plutonium (III) Diode Array Spectrophotometry¹

This standard is issued under the fixed designation C1307; 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 describes the determination of total plutonium as plutonium(III) in nitrate and chloride solutions. The technique is applicable to solutions of plutonium dioxide powders and pellets (Test Methods C697), nuclear grade mixed oxides (Test Methods C698), plutonium metal (Test Methods C758), and plutonium nitrate solutions (Test Methods C759). Solid samples are dissolved using the appropriate dissolution techniques described in Practice C1168. The use of this technique for other plutonium-bearing materials has been reported (1-5), but final determination of applicability must be made by the user. The applicable concentration range for plutonium sample solutions is 10–200 g Pu/L.²

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

1.3 *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: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)O₂)

C757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable

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

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets

C859 Terminology Relating to Nuclear Materials

C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms related to nuclear materials, refer to Terminology C859.

4. Summary of Method

4.1 In a diode array spectrophotometric measurement, as in a conventional spectrophotometric measurement, the substance being determined absorbs light at frequencies characteristic of that substance. The amount of light absorbed at each wavelength is directly proportional to the concentration of the species of interest. The absorption is a function of the oxidation state and the complexation obtained in the solution matrix selected for measurement. Beer's Law permits quantifying the species of interest relative to a traceable standard when both solutions are measured under the same conditions. The array of photosensitive diodes permits the (virtually) simultaneous collection of spectral information over the entire range of the instrument, for example, 190–820 nm (or any selected portion of the range). An entire absorption spectrum can be obtained in 0.1 s; however, optimum precision is obtained from multiple spectra collected over a 4-s period.

4.2 Reduction to plutonium(III) is accomplished by the addition of a measured quantity of reductant solution to the sample aliquant.

4.2.1 For nitrate solutions, ferrous sulfamate is the recommended reductant. Aliquants (1 mL or less) of the sample

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, 2015. Published January 2015. Originally approved in 1995. Last previous edition approved in 2014 as C1307 – 14. DOI: 10.1520/C1307-15.

² For solid samples, select the sample size and dissolved solution weight to yield sample solutions in the 10–30 g Pu/L range. With special preparation and spectral analysis techniques, the method has been applied to nitrate solutions in the 0.1–10 g Pu/L range.

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.