



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

This standard is issued under the fixed designation C 1307; 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 C 697), nuclear grade mixed oxides (Test Methods C 698), plutonium metal (Test Methods C 758), and plutonium nitrate solutions (Test Methods C 759). Solid samples are dissolved using the appropriate dissolution techniques described in Practice C 1168. 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 *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:

- C 697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets³
- C 698 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Mixed Oxides ((U, Pu)O₂)³
- C 757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable³
- C 758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal³
- C 759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of

Nuclear-Grade Plutonium Nitrate Solutions³

C 833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets³

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis³

3. Summary of Method

3.1 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.

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

3.2.1 For nitrate solutions, ferrous sulfamate is the recommended reductant. Aliquants (1 mL or less) of the sample solution are diluted with 10 mL of a ferrous reductant/matrix solution to 1 g Pu/L, and measured.

3.2.2 For chloride solutions, ascorbic acid is the recommended reductant. Aliquants of the sample solution, each containing 50–100 mg of plutonium, are diluted with 2 mL of zirconium solution to complex fluoride ions, 2 mL ascorbic acid reductant solution, and 1.0 M HCl to a total volume of 25 mL, yielding 2–4 g Pu/L solutions for measurement.

3.3 Plutonium concentration is determined from light absorption measurements taken on the sample solution in the blue-green region from 516 to 640 nm where a broad doublet band is observed. Spectral quantifying software capable of fitting the sample spectrum with spectral information from standard solutions is used to calculate the plutonium concentration. Both commercially available (6) and custom-designed

¹ This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved Jan. 10, 2002. Published May 2002. Originally published as C1307–95. Last previous edition C1307–95.

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 *Annual Book of ASTM Standards*, Vol 12.01.