



Standard Test Method for Determining Plutonium by Controlled-Potential Coulometry in H₂SO₄ at a Platinum Working Electrode¹

This standard is issued under the fixed designation C 1165; 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.

^{ε1} NOTE—Section 2 and footnote 6 were editorially updated in July 2000.

1. Scope

1.1 This test method describes the determination of milligram quantities of plutonium in unirradiated uranium-plutonium mixed oxide having a U/Pu ratio range of 0.1 to 10. This test method is also applicable to plutonium metal, plutonium oxide, uranium-plutonium mixed carbide, various plutonium compounds including fluoride and chloride salts, and plutonium solutions.

1.2 The recommended amount of plutonium for each aliquant in the coulometric analysis is 5 to 10 mg. Precision worsens for lower amounts of plutonium, and elapsed time of electrolysis becomes impractical for higher amounts of plutonium.

1.3 The values stated in SI units are to be regarded as 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. Specific precautionary statements are given in Section 8.*

2. Referenced Documents

2.1 ASTM Standards:

- C 757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable²
- C 758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal²
- C 759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions²
- C 833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets²
- C 859 Terminology Relating to Nuclear Materials²
- C 1009 Guide for Establishing a Quality Assurance Pro-

gram for Analytical Chemistry Laboratories Within the Nuclear Industry²

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry²

C 1108 Test Method for Plutonium by Controlled-Potential Coulometry²

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials²

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials²

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis²

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry²

C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials²

3. Summary of Test Method

3.1 In controlled-potential coulometry, the analyte reacts at an electrode having a maintained potential that precludes reactions of as many impurity components as is feasible. In the electrolysis, current decreases exponentially as the reaction proceeds until a selected background current is reached. The quantity of analyte reacted is calculable by Faraday's law. Detailed discussions of the theory and applications of this technique are presented in Refs (1)³ and (2).

3.2 Plutonium and many impurity element ions are initially reduced in a 0.5 M H₂SO₄ electrolyte at a platinum working electrode (3) maintained at +0.310 V versus a saturated calomel electrode (SCE). Plutonium is then oxidized to Pu(IV) at a potential of +0.670 V. The quantity of plutonium is calculated from the number of coulombs required for oxidation according to Faraday's law.

$$Q = \int_0^t i \, dt = n w F / M \quad (1)$$

Rearrangement to solve for w gives:

$$w = M Q / n F \quad (2)$$

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.

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² Annual Book of ASTM Standards, Vol 12.01.

³ The boldface numbers in parentheses refer to a list of references at the end of the text.