



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

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1. Scope

1.1 This test method covers 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. 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.* Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:²

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

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.

Nuclear-Grade Plutonium Nitrate Solutions

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets

C859 Terminology Relating to Nuclear Materials

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1108 Test Method for Plutonium by Controlled-Potential Coulometry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry

C1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

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

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