



Standard Test Method for Plutonium by Controlled-Potential Coulometry¹

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1. Scope

1.1 This test method describes the determination of plutonium in solutions of unirradiated nuclear-grade (that is, high-purity) materials by controlled-potential coulometry. Controlled-potential coulometry may be performed in a choice of supporting electrolytes, such as 0.9 M HNO₃, 1 M HClO₄, 1 M HCl, 5 M HCl, and 0.5 M H₂SO₄. Limitations on the use of selected supporting electrolytes are discussed in Section 5. Optimum quantities of plutonium for this procedure are 5 to 10 mg.

1.2 Plutonium-bearing materials are radioactive and toxic. Adequate laboratory facilities, such as gloved boxes, fume hoods, controlled ventilation, etc., along with safe techniques must be used in handling specimens containing these materials.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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:

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry²

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry³

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials³

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials³

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

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 A Julie 100-Ω precision resistor number NB102A, accurate to 0.0015 %, has been found satisfactory.

³ Annual Book of ASTM Standards, Vol 12.01.

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry³

C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

3. Summary of Test Method

3.1 In a controlled-potential coulometric measurement, the substance being determined reacts at an electrode, the potential of which is maintained at such a value that unwanted electrode reactions are precluded under the prevailing experimental conditions. Those substances which have reduction-oxidation (redox) potentials near that of the ion being determined constitute interferences. Electrolysis current decreases exponentially as the reaction proceeds, until constant background current is obtained. Detailed discussions of the theory and applications of this technique have been published (1, 2, 3, 4, 5, 6).⁵ The control-potential adjustment technique (7) can be used to terminate the electrolysis of the specimen at constant background current without exhaustive electrolysis with considerable reduction in operating time. Use of the control-potential adjustment technique requires that the coulometer integrator be capable of operations in a bipolar mode and that the plutonium-containing solution be of high purity, that is, nuclear grade.

3.2 Plutonium(IV) is reduced to Pu(III) at a working electrode maintained at a potential more negative than the formal redox potential. Plutonium(III) is oxidized to Pu(IV) at a potential more positive than the formal redox potential. The quantity of plutonium electrolyzed is calculated from the net number of coulombs required for the electrolysis, according to Faraday's law. Corrections for incomplete reaction, derived from the Nernst equation, must be applied for electrolysis of the sample aliquot (7, 8).

$$W = \frac{(Q_s - Q_b) M}{nFf} \quad (1)$$

where:

W = grams of plutonium,

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ The boldface numbers in parentheses refer to the list of references at the end of this test method.