



Standard Test Method for Plutonium by Titanium(III)/Cerium(IV) Titration¹

This standard is issued under the fixed designation C 1235; 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 is applicable to the assay or purity determination of plutonium metal of 98 % purity or higher. Uranium and iron are known interferences and must be determined separately. Their respective corrections must then be made to the assay value.

1.2 The recommended amount of plutonium determined in the titration is 210 to 240 mg.

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:

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 Industry²

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 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 a redox titration, as titrant is added, the change in concentration of the redox couple is monitored. This change in concentration of the redox couple can be monitored by measuring the potential difference between a platinum indicator electrode and a reference electrode (such as a saturated

calomel electrode) in contact with the solution or by other equivalent methods of endpoint determination. The endpoint of the titration is usually chosen to be the point at which the rate of change in concentration of the redox couple is greatest per increment of titrant added. The concentration of analyte is calculated from the volume or mass of titrant added to reach the endpoint, the concentration of the titrant, and the mass of the sample titrated. Lingane (1)³ discusses the principles of automatic potentiometric titrations, and Meites (2) lists numerous examples of potentiometric redox titrations.

3.2 This test method is an adaptation of the Coppel and Regnaud method (3) in which the plutonium in solution is reduced to Pu(III) with titanium(III) chloride, excess Ti(III) is destroyed with nitric acid, and finally, the reduced plutonium is oxidized to Pu(IV) with ceric titrant using ferroin indicator. However, this adaptation substitutes a potentiometric endpoint for the indicator endpoint and uses commercial titration instrumentation (4, 5).

3.3 This test method was developed for production support and has distinct advantages when a large number of samples are to be analyzed. It is largely automated, accomplishing a titration approximately every 2 min when optimizing step-wise operations. Variations on this test method, such as manual titration with visual or photometric endpoint detection or use of a weight buret would, no doubt, provide quality data, but at the expense of limiting sample throughput.

4. Significance and Use

4.1 This test method is designed to determine the plutonium content of plutonium metal.

4.2 Committee C-26 Safeguards Statement⁴:

4.2.1 The material (plutonium metal) to which this test method applies is subject to nuclear safeguards regulations governing its possession and use. Materials for use by the commercial nuclear community must also meet compositional specifications.

4.2.2 The analytical method in this test method both meets U.S. Department of Energy guidelines for acceptability of a measurement method for generation of safeguards accountability measurement data and also provides data that may be used

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 the list of references at the end of this test method.

⁴ Based upon Committee C-26 Safeguards Matrix (C 1009, C 1068, C 1128, C 1156, C 1210, and C 1297).