



Standard Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium¹

This standard is issued under the fixed designation C1267; 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, commonly referred to as the Modified Davies and Gray technique, covers the titration of uranium in product, fuel, and scrap materials after the material is dissolved. The test method is versatile and has been ruggedness tested. With appropriate sample preparation, this test method can give precise and unbiased uranium assays over a wide variety of material types (**1**, **2**).² Details of the titration procedure in the presence of plutonium with appropriate modifications are given in Test Method **C1204**.

1.2 Uranium levels titrated are usually 20 to 50 mg, but up to 200 mg uranium can be titrated using the reagent volumes stated in this test method.

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. For specific safeguard and safety precaution statements, see Section 4.*

2. Referenced Documents

2.1 ASTM Standards:³

C696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets

C799 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of

Nuclear-Grade Uranyl Nitrate Solutions

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

C1204 Test Method for Uranium in Presence of Plutonium by Iron(II) Reduction in Phosphoric Acid Followed by Chromium(VI) Titration

C1346 Practice for Dissolution of UF₆ from P-10 Tubes⁴

C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis

2.2 NIST Standard:⁴

SRM 136e Potassium Dichromate (Oxidimetric Standard)

2.3 NBL Standard:⁵

CRM 112A Uranium Metal Standard

3. Summary of Test Method

3.1 Samples are prepared by dissolution techniques detailed in Practices **C1346**, **C1347**, or Refs (**2**), (**3**), and (**4**). Appropriate uncertainties for sampling and weight determination should be applied to the overall precision and bias calculations for the final result. Aliquants containing 20 to 200 mg of uranium are prepared by weight. The sample is fumed to dryness after the appropriate acid treatment. The sample is dissolved in dilute nitric acid or water prior to titration.

3.2 Uranium is reduced to uranium(IV) by excess iron(II) in concentrated phosphoric acid (H₃PO₄) containing sulfamic acid. The excess iron(II) is selectively oxidized by nitric acid (HNO₃) in the presence of a molybdenum(VI) catalyst. After the addition of a vanadium(IV) solution, the uranium(IV) is titrated with chromium(VI) to a potentiometric end point.

3.3 The chromium(VI) titrant may be delivered manually on a weight or on a volumetric basis as specified by the facility titration procedure.

3.3.1 If the titrant is delivered on a volumetric basis, corrections to the volume of titrant may be needed to adjust for the difference between the temperature of preparation and the ambient temperature.

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 The boldface numbers in parentheses refer to the list of references at the end of this standard.

³ 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.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

⁵ New Brunswick Laboratory (NBL) Certified Reference Materials Catalog (U.S. Department of Energy), <http://www.nbl.doe.gov>.