



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 C 1267; 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 C 1204.

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 *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:

C 696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets³

C 799 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions³

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

C 1204 Test Method for Uranium in the Presence of Plutonium by Iron(II) Reduction in Phosphoric Acid Followed by Chromium(VI) Titration³

C 1346 Practice for Dissolution of UF₆ from P-10 Tubes³

C 1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis³

3. Summary of Test Method

3.1 Samples are prepared by dissolution techniques detailed in Practices C 1346, C 1347, 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.

3.3.2 Automated titrators are facility specific and are not explicitly addressed in this test method. However, automated titrators which have comparable bias and precision may be used.

3.3.3 There is an alternate, high precision (~0.005 % RSD) modified Davies and Gray titration, which is similar to the method covered in this procedure except that the amount of uranium titrated is increased and about 90 % of the titrant is delivered on a solid weight basis followed by titration to the end point with a dilute titrant. Details of this alternate procedure are available in Ref (5).

4. Significance and Use

4.1 Factors governing selection of a method for the determination of uranium include available quantity of sample, homogeneity of material sampled, sample purity, desired level of reliability, and facility available equipment.

4.2 This uranium assay method is referenced in the Test

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

³ Annual Book of ASTM Standards, Vol 12.01.