



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

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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>.

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 ($\sim 0.005\%$ RSD) modified Davies and Gray titration, which is similar to the method covered in this procedure. In the high precision method, 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 method 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 Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets (Test Methods C696) and in the Test Methods for Chemical, Mass Spectrometric, and Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions (Test Methods C799). This uranium assay method may also be used for uranium hexafluoride and uranium ore concentrate. This test method determines 20 to 200 mg of uranium; is applicable to product, fuel, and scrap material after the material is dissolved; is tolerant towards most metallic impurity elements usually specified in product and fuel; and uses no special equipment.

4.3 The ruggedness of the titration method has been studied for both the volumetric (6) and the weight (7) titration of uranium with dichromate.

4.4 Committee C26 Safeguards Statement:

4.4.1 The materials (nuclear grade uranium in product, fuel, and scrap) to which this test method applies are subject to nuclear safeguard regulations governing their possession and use. The analytical method in this standard meets U.S. Department of Energy guidelines for acceptability of a measurement method for generation of safeguards accountability measurement data.

4.4.2 When used in conjunction with the appropriate certified reference materials (SRM⁶ or CRM), this procedure can demonstrate traceability to the national measurement base. However, use of the test method does not automatically guarantee regulatory acceptance of the resulting safeguards measurements. It remains the sole responsibility of the user of this test method to assure that its application to safeguards has the approval of the proper regulatory authorities.

5. Interferences

5.1 Interfering elements are not generally present in product and fuel material in quantities which cause interference in the titration.

5.1.1 Of the metallic impurity elements usually included in specifications for product and fuel, silver, manganese, and vanadium (in the V oxidation state) interfere when present in amounts of 10 mg or greater of impurity per 100 mg of uranium (2, 8).

5.1.2 Silver and vanadium (in the V oxidation state) cause positive bias when present in milligram quantities in the sample. The aliquant treatment adjusts the oxidation state of any vanadium(V) present in the sample (2). To remove silver, the sample must be treated prior to titration (8).

5.1.3 Manganese was originally found to cause a negative bias (2), but this bias is eliminated when the titration aliquant preparation procedure is followed as given (9, 10) in this titrimetric method.

5.2 Interferences with the Modified Davies and Gray titration, which may be present in some uranium materials, have been systematically studied.

5.2.1 The non-interference of copper, titanium, cobalt, nickel, cerium, and samarium was demonstrated (11) at the 50 mg impurity level for 100 mg of uranium.

5.2.2 The effects of the following elements in milligram quantities were studied: silver, gold, lead, iodine, arsenic, antimony, and bismuth (8).

5.2.2.1 Gold, lead, arsenic(V), antimony(V), and bismuth do not interfere when present in amounts of 10 mg for 100 mg of uranium.

5.2.2.2 Silver, iodine, arsenic(III), and antimony(III) interfere seriously in the determination of uranium and must be eliminated prior to titration.

5.2.3 The effects of impurities on the titration of uranium continued with the platinum metals (ruthenium, rhodium, palladium, osmium, iridium, and platinum), chloride, bromide (12), fluoride (13), and technetium (14).

5.2.3.1 Ruthenium, palladium, osmium, iridium, and platinum cause serious positive errors in the determination of uranium. Rhodium alone among the platinum metals does not cause any significant error.

5.2.3.2 Chloride and bromide interfere with the assay through their effect on the platinum indicator electrode.

5.2.3.3 Small amounts of fluoride, less than 400 mg as hydrofluoric acid (HF) or 600 mg if HNO₃ is present, can be tolerated by the titration.

5.2.3.4 Technetium, found in high temperature reactor grade recycle (htgr) fuel, interferes with the titration and must be removed before titration.

5.3 The removal of certain interferences in the modified Davies and Gray titration has also been studied.

5.3.1 The initial fuming of titration aliquants with sulfuric acid removes impurity elements such as the halides and volatile metallic elements (2, 12, 13).

5.3.2 Arsenic(III) and antimony(III) can be eliminated in the H₃PO₄ by potassium dichromate (K₂Cr₂O₇) oxidation prior to its addition to the titration medium (8).

5.3.3 Elimination of interferences in the titration by mercury, platinum, and palladium by means of a copper column was evaluated (15).

5.3.4 Elimination of interferences by solvent extraction of the uranium from the impurities has also been studied (16).

⁶ SRM is a registered trademark.

5.4 A list of impurities with brief references to their treatment for elimination is given in Table A1.1 in Annex A1, and the details are given in Refs 2, 8, 9, 10, and 12-16.

6. Apparatus

6.1 *Buret*, polyethylene bottle (preparation instructions can be found in Appendix X1), glass weight, or glass volumetric.

6.2 *pH Meter*, with indicator (a 16-gauge platinum wire has been found to be satisfactory) and reference (saturated calomel has been found to be satisfactory) electrodes.

6.2.1 The indicator electrode should be changed or cleaned if there is a titration problem such as less distinct than normal end point break or end point drift, or, if desired, prior to use when more than a week has passed since its last use. Suggested cleaning procedures for platinum wire electrodes are detailed in Appendix X2.

6.2.2 Asbestos and glass bead tipped saturated calomel electrodes can be placed directly in the titration solution. Glass frit tipped saturated calomel electrodes may have a faster leak rate and may need to be used with a separator tube containing the electrolyte to prevent titration problems due to chloride.

6.2.3 The reference electrode should be covered with a rubber tip or submerged in a solution (saturated potassium chloride solution for the calomel electrode) for overnight storage.

6.3 *Magnetic Stirrer and TFE-Fluorocarbon Coated Magnet*.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁷ Other grades of reagents may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean laboratory accepted demineralized or deionized water.

7.3 *Ferrous Sulfate Heptahydrate* ($\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$, 1.0 M)—Add 100 mL of sulfuric acid (H_2SO_4 , sp gr 1.84) to 750 mL of water as the solution is stirred. Add 280 g of $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$, and dilute the solution to 1 L with water. Prepare the $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ reagent fresh, weekly. See the Note 5 in 10.8 on combination of this reagent with the H_3PO_4 .

7.4 *Nitric Acid* (HNO_3 , 8 M)—Add 500 mL of HNO_3 (sp gr 1.42) to <500 mL of water, and dilute to 1 L.

7.5 HNO_3 , 1 M—Add 64 mL of HNO_3 (sp gr 1.42) to <900 mL of water, and dilute to 1 L.

7.6 HNO_3 (8 M)—Sulfamic Acid ($\text{NH}_2\text{SO}_3\text{H}$, 0.15 M)—Ammonium Molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, 0.4 %)—Dissolve 4 g of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ in 400 mL of water, and add 500 mL of HNO_3 (sp gr 1.42). Mix and add 100 mL of 1.5 M $\text{NH}_2\text{SO}_3\text{H}$ solution (see 7.10) and mix.

7.7 *Orthophosphoric Acid* (H_3PO_4), 85 %—Test for reducing substances prior to use (see Annex A2).

7.8 *Potassium Dichromate* ($\text{K}_2\text{Cr}_2\text{O}_7$) Solution (2 %)—Dissolve 2 g of $\text{K}_2\text{Cr}_2\text{O}_7$ in water, and dilute to 100 g with water.

7.9 $\text{K}_2\text{Cr}_2\text{O}_7$ (0.0045 M)—Dissolve 2.65 g of reagent grade or purer grade $\text{K}_2\text{Cr}_2\text{O}_7$ in water; transfer this solution to a pre-weighed, 2-L volumetric flask or suitable alternative and dilute to volume, or use equivalent weight/volume ratios for larger quantities of solution.

7.9.1 If National Institute of Standards and Technology (NIST) standard reference material $\text{K}_2\text{Cr}_2\text{O}_7$ (SRM 136e or its equivalent) was used, proceed as in 7.9.1.1 and 7.9.1.2 before going to 7.9.3; otherwise go to 7.9.2.

7.9.1.1 Allow the solution to equilibrate to room temperature, obtain the weight of the solution. Compute the dichromate concentration (11.2.2) and the uranium titration factor (11.3.2) after correcting the weight of dichromate for buoyancy (11.1.2) and for purity (11.1.3).

7.9.1.2 As a good quality practice, a check on the material handling of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution within laboratory accepted uncertainties may be done by titration with a working reference uranium solution. For guidance in the preparation of a working reference uranium solution, see Guide C1128. If the titrations do not agree within laboratory accepted uncertainties, verification titrations for SRM 136e or its equivalent may be done using New Brunswick Laboratory (NBL) CRM 112-A uranium metal or its equivalent,⁵ prepared in 7.12, or the $\text{K}_2\text{Cr}_2\text{O}_7$ solution should be discarded.

7.9.2 If a reagent grade $\text{K}_2\text{Cr}_2\text{O}_7$ was used, allow the solution to equilibrate to room temperature and standardize the $\text{K}_2\text{Cr}_2\text{O}_7$ solution against CRM 112-A uranium metal or its equivalent prepared in 7.12 (see Appendix X3). Compute the uranium titration factor as in 11.3.3.

7.9.3 Store the $\text{K}_2\text{Cr}_2\text{O}_7$ solution in one or more borosilicate glass bottles with a poly-seal top or an equivalent container to prevent concentration changes due to evaporation.

7.10 $\text{NH}_2\text{SO}_3\text{H}$ (1.5 M)—Dissolve 146 g of $\text{NH}_2\text{SO}_3\text{H}$ in water, filter the solution, and dilute to 1 L.

7.11 *Sulfuric Acid* (H_2SO_4 , 1 M)—Add 56 mL of H_2SO_4 (sp gr 1.84) to water, while stirring, and dilute to 1 L with water.

7.12 *Uranium Standard (CRM) Solution*:

7.12.1 Clean the surface of the uranium metal (CRM 112-A or its equivalent) following the instructions on the certificate.

7.12.2 Obtain the weight of the metal by difference to 0.01 mg making buoyancy and purity corrections detailed in 11.1.2 and 11.1.3, respectively.

7.12.3 Prepare the uranium standard solution. There are many successful methods of uranium metal dissolution (none is specified on the CRM 112-A certificate); methods which

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

reproduce the uranium assay value on the certificate are acceptable. An example of an acceptable method is given in [Appendix X4](#).

7.12.4 Equilibrate the uranium solution to room temperature, and obtain the weight of the solution to give the same number of significant figures as the metal's weight.

7.12.4.1 Calculate the solution concentration in mg U/g uranium solution using the calculation in [11.2.3](#).

7.12.4.2 As a good quality practice, a check on the material handling of the uranium solution within laboratory accepted uncertainties may be done by titration with a working reference $K_2Cr_2O_7$ solution. For guidance in the preparation of a working reference $K_2Cr_2O_7$ solution, see [Guide C1128](#). If the titrations do not agree within laboratory accepted uncertainties, verification titrations for CRM 112-A or its equivalent may be done using $K_2Cr_2O_7$ SRM 136e or its equivalent, as prepared in [7.9](#), or the uranium solution should be discarded.

7.13 *Vanadyl Sulfate Dihydrate*—($VOSO_4 \cdot 2H_2O$) Solution (0.0038 M—0.18 M H_2SO_4)—Add 20 mL concentrated H_2SO_4 (sp gr 1.84) to <980 mL water with stirring; equilibrate to room temperature. Weigh 1.5 g of $VOSO_4 \cdot 2H_2O$ crystals, mix the solid with the temperature equilibrated H_2SO_4 solution, and dilute the solution to 2 L. The $VOSO_4 \cdot 2H_2O$ concentration should provide 75 to 125 mg $VOSO_4 \cdot 2H_2O$ per titration, but the concentration is not critical. See Refs [\(6\)](#) and [\(7\)](#).

7.13.1 The $VOSO_4 \cdot 2H_2O$ solution is not stable [\(17\)](#); H_2SO_4 stabilizes the V(IV) oxidation state, but the H_2SO_4 concentration is not critical. The $VOSO_4 \cdot 2H_2O$ solution should be prepared at suitable intervals to prevent V(V) interference (24 h intervals for preparation are suggested) since this solution is added after the aliquant treatment stage.

7.13.2 Alternatively, crystalline $VOSO_4 \cdot 2H_2O$ (75 to 125 mg per titration) may be used with a water diluent in place of the solution (see [10.13](#)).

NOTE 1—There is more than one crystalline form of vanadyl sulfate; there are problems with slow dissolving forms particularly when the vanadyl sulfate is added as a solid. Since vendors are now selling vanadyl sulfate with an unspecified hydration number, that is, $VOSO_4 \cdot xH_2O$, note that the material should be bright blue in color and have a wet appearance. The difficult-to-dissolve crystalline form is light blue and dry in appearance.

8. Hazards

8.1 Since enriched uranium-bearing materials are radioactive and toxic, adequate laboratory facilities and fume hoods along with safe techniques must be used in handling samples containing these materials. A detailed discussion of all precautions necessary is beyond the scope of this test method. However, personnel who handle radioactive materials should be familiar with the safe handling practices of the facility.

8.2 In the absence of data about the bioavailability of the chromium(VI) in $K_2Cr_2O_7$,⁸ the facility policy for handling of $K_2Cr_2O_7$ should be followed. In some facilities $K_2Cr_2O_7$ has been identified as a “select carcinogen” as a conservative policy.

⁸ Overall Evaluations of Carcinogenicity: An Updating of International Agency for Research on Cancer (IARC) Monographs, Supplement 7, World Health Organization, International Agency for Research on Cancer, 1987, p. xvii.

8.3 **Warning**—Hydrofluoric acid is highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature, and the duration of contact with the acid. Hydrofluoric acid differs from the other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Due to the serious consequences of hydrofluoric acid burns, prevention of exposure or injury of personnel is the primary goal. Utilization of appropriate laboratory controls (hoods) and wearing adequate personnel protective equipment to protect from skin and eye contact is essential. Acute exposure to HF can cause painful and severe burns upon skin contact that require special medical attention. Chronic or prolonged exposure to low levels on the skin may cause fluorosis.

9. Calibration

9.1 Traceability to a national or international measurement base may be demonstrated by the use of standard reference materials, for example, NIST standard SRM 136e or its equivalent (potassium dichromate), or NBL standard CRM 112-A (uranium metal standard).

9.2 The standard $K_2Cr_2O_7$ should be prepared as instructed on the certificate; the weight should be obtained to 0.01 mg and corrected for buoyancy and purity using the calculations in [11.1.2](#) and [11.1.3](#).

9.2.1 If a $K_2Cr_2O_7$ solution is prepared from the solid $K_2Cr_2O_7$, the solution should be equilibrated to room temperature, and the weight should be obtained to give a number of significant figures equal to that of the solid's weight.

9.2.2 The $K_2Cr_2O_7$ solution concentration is calculated in mg $K_2Cr_2O_7$ /g $K_2Cr_2O_7$ solution using the calculation in [11.2.2](#). The titration factor (mg uranium/g $K_2Cr_2O_7$ solution) is calculated for the $K_2Cr_2O_7$ solution using the calculation in [11.3.2](#).

9.3 If reagent grade $K_2Cr_2O_7$ is used, the material must be standardized against the NBL uranium metal standard (CRM 112-A or its equivalent) to provide traceability to the national measurement base.

NOTE 2—**Caution:** Satisfactory analysis results will only be attained if the temperature of the reagents (usually at room temperature) used are within the range of 23 to 31°C.

9.3.1 Analyze individually dispensed aliquants of the uranium reference solution per [10.3-10.14.4](#). See [Appendix X3](#) for analysis control recommendations.

9.3.2 Calculate the uranium titration factor (mg U/g $K_2Cr_2O_7$ solution) for the standardized $K_2Cr_2O_7$ solution using the calculation in [11.3.2](#).

10. Titration Procedure

NOTE 3—**Caution:** Satisfactory analysis results will only be attained if the temperature of the reagents (usually at room temperature) used are within the range of 23 to 31°C.

10.1 Obtain the weight of the sample (0.5 g or more) to 0.1 mg. When necessary, especially for oxides, include errors due

to weight determination in the calculation of the overall measurement uncertainty. Dissolve the sample following the procedures in Practices C1346, C1347, or Refs 2-4.

10.2 To prepare aliquants, quantitatively transfer the dissolved sample with an accurately determined weight to a bottle with an accurately determined weight for mixing prior to sample splitting.

10.2.1 A low-density polyethylene narrow mouth bottle with a one-piece polypropylene seal-ring screw closure to prevent leakage, or any other leak-proof bottle may be used. If polyethylene bottles are used, long-term (weeks and months) storage will not maintain sample integrity because of bottle respiration (18).

10.2.2 Equilibrate the solution to room temperature, and weigh the solution to the number of significant figures equivalent to the sample's weight.

10.2.3 Invert and shake the solution to insure a homogeneous solution prior to aliquanting.

10.2.4 Calculate the sample dilution factor (g sample/g solution).

10.3 Deliver an aliquant, with a weight determined to 0.1 mg accuracy, containing 20 to 100 mg of uranium, into the titration vessel (400-mL beakers have been found to be satisfactory).

10.4 Add 1 mL of 1 M H₂SO₄ to the aliquant, unless the sample contains elements which form insoluble sulfates, for example, calcium and aluminum, and fume to dryness. For specific elemental interference treatments, see Annex A1 and the references cited therein.

NOTE 4—The maximum acid contents (2, 19) for a sample aliquant to be analyzed by this method are 4 mL H₂SO₄ (sp gr 1.84), 3 mL HNO₃ (sp gr 1.42), 5 mL HClO₄ (sp gr 1.67), no HCl, and 0.5 mL free (that is, not complexed such as with zirconium in U-Zr alloys) HF (sp gr 1.17). Aliquants which are fumed to dryness with sulfuric acid should not require further treatment to satisfy these requirements.

10.5 Dissolve the sample in 15 mL of water or in 15 mL of 1 M HNO₃ using the water or reagent to rinse down the sides of the beaker. The total dissolution of the sample at this point is critical to accurate analysis; a wait of 30 min to 1 h is recommended to insure total dissolution.

10.6 Add 5 mL of 1.5 M NH₂SO₃H to the beaker, and mix by swirling. Use this solution to rinse the sides of the beaker.

10.7 Add 40 mL of H₃PO₄ (85 %), treated with dichromate (see Annex A2), directly into the sample. The splashing of any solution onto the side of the beaker should be avoided.

10.8 Add 5 mL of FeSO₄·7 H₂O solution, and swirl briefly. Do not allow the FeSO₄·7 H₂O solution to touch the sides of the beaker while dispensing this reagent.

NOTE 5—The FeSO₄·7 H₂O solution may be combined with the H₃PO₄ in 10.7 and added as a combined reagent.

10.9 Add a TFE-fluorocarbon coated magnet without splashing, place the beaker on a magnetic stirrer, and initiate stirring at a slow rate (avoid splashing) for 30 s.

10.10 Add 10 mL of nitric-sulfamic-molybdate solution. Use the solution to rinse down the sides of the beaker.

10.11 Mix the solution at a moderate stirrer speed. Immediately upon disappearance of the black color, begin timing the oxidation period of 3 min; the solution may or may not be stirred during the 3 min time period.

10.12 Obtain the weight of the dichromate in the weight buret if a gravimetric titration is to be used; otherwise, zero the buret.

10.13 Stop the stirring, add 100 mL of the VOSO₄·2H₂O solution, or add the diluent (water) if crystalline VOSO₄·2H₂O is used.

10.13.1 If crystalline VOSO₄·2H₂O (75 to 125 mg) is used, add it after the diluent.

10.13.2 Use the VOSO₄·2H₂O solution or diluent to rinse the sides of the beaker.

10.14 Increase the rate of stirring to form a vortex in the solution.

10.14.1 Insert the electrodes into the solution, and titrate rapidly with K₂Cr₂O₇ to a potential of 450 to 480 mV versus a calomel reference electrode or to the equivalent voltage for other reference electrodes. If the polyethylene weight buret is used, remove the reduced size tip used in the final end point approach before beginning the addition of K₂Cr₂O₇ solution.

10.14.2 Decrease the rate of K₂Cr₂O₇ solution additions to large drops, one to two drop portions; titrate to a potential of 500 mV or to the equivalent for reference electrodes other than calomel.

10.14.3 Begin smaller K₂Cr₂O₇ solution drop-size additions (for the polyethylene weight buret, place the micro-tip on the weight buret), and titrate to the potential break, or if a second derivative technique is to be used, skip to 10.14.4.

10.14.3.1 The maximum time elapsed between the addition of the VOSO₄·2H₂O solution or diluent and the completion of 99 + % of the titration should not exceed 5 min.

10.14.3.2 The best precision will be attained if the time is limited to 3 to 5 min elapsed time.

10.14.3.3 The variation in the final potential readings to maintain acceptable precision should be 590 mV ± 20 mV or equivalent potentials for reference electrodes other than the calomel.

10.14.4 If a double derivative end point is used instead of a fixed end point, titrate near the potential break (550 to 580 mV or equivalent) using small drops and recording each buret and potential reading. Record one drop reading past the end point, and calculate the end point using a double derivative technique.

10.14.4.1 The precautions in 10.14.3.1 and 10.14.3.2, regarding the time limits for the titration, apply up to completion of 99 + % of the titration.

10.14.4.2 The double derivative end point approach may require more than 5 min, but since 99 + % of the uranium has been titrated, the additional time will not significantly affect the final results.

10.14.4.3 Better precision will be obtained if the total titration time is limited to 10 min or less.

10.14.5 Alternative end point procedures used in manual or automated titration systems, which have been demonstrated to give comparable accuracy, are also acceptable.

11. Calculation

11.1 Buoyancy and Purity Corrections:

11.1.1 When NIST standard $K_2Cr_2O_7$ (SRM 136e or equivalent) is used for standard solution preparation, corrections for buoyancy and purity should be applied to the solid material's weight. If NBL standard uranium metal (CRM 112-A or equivalent) is used to prepare a standard uranium solution, corrections for buoyancy and purity should be applied to the metal's weight.

11.1.2 The buoyancy correction is made using the following formula:

$$W_v = W_o[1 + (1/D_o - 1/D_w)D_a] \quad (1)$$

where:

W_v = weight of the object in vacuum, g,
 W_o = weight of the object in air, g,
 D_o = density of the object in air,
 D_w = density of the weights of the balance in air, and
 D_a = density of air at the temperature and pressure at which the weight of the object was determined.

11.1.3 The purity correction is made using the following formula:

$$W_c = (W_v)(PF) \quad (2)$$

where:

W_c = corrected weight of material, g,
 W_v = buoyancy corrected weight of material, g, and
 PF = purity factor stated on certificate, %/100.

11.2 Concentration Calculations:

11.2.1 Calculations of concentrations for standard solutions of $K_2Cr_2O_7$ and of uranium are made using the buoyancy and purity corrected weight for the solids.

11.2.2 The concentration of the standard $K_2Cr_2O_7$ solution is calculated using the following equation:

$$C_c = (D_c)/L \quad (3)$$

where:

C_c = concentration of $K_2Cr_2O_7$, mg $K_2Cr_2O_7$ /g $K_2Cr_2O_7$ solution,
 D_c = corrected weight of $K_2Cr_2O_7$ solid in mg from 11.1.3 for $K_2Cr_2O_7$, (1000 mg/g) W_c , and
 L = $K_2Cr_2O_7$ solution weight, g $K_2Cr_2O_7$ solution.

11.2.3 The concentration of the standard uranium solution is calculated using the following equation:

$$C_u = (D_u)/Q \quad (4)$$

where:

C_u = concentration of uranium solution, mg uranium/g uranium solution,
 D_u = corrected weight of uranium metal in mg from 11.1.3 for uranium metal, (1000 mg/g) W_c , and
 Q = standard uranium solution weight, g uranium solution.

11.3 Uranium Titration Factor:

11.3.1 The titration factor is calculated in mg uranium/g $K_2Cr_2O_7$ solution.

11.3.2 For the standard $K_2Cr_2O_7$ solution, the uranium titration factor is calculated from the $K_2Cr_2O_7$ concentration factor and is based on the reaction of $K_2Cr_2O_7$ with uranium(IV), that is



Since 3 moles of uranium(IV) react with 1 mole of $K_2Cr_2O_7$, the equivalent multiplier for the $K_2Cr_2O_7$ to uranium conversion is illustrated as follows:

$$\frac{(\text{molecular weight uranium}) (3)}{(\text{molecular weight } K_2Cr_2O_7)} = 2.42734 \quad (5)$$

for CRM 112-A (238.0289 g/mole) and $K_2Cr_2O_7$ (294.1844 g/mole). The uranium titration factor (mg U/g $K_2Cr_2O_7$ solution) is calculated for the standard $K_2Cr_2O_7$ solution concentration using the following equation:

$$T = (C_c)(M) \quad (6)$$

where:

T = titrant factor for $K_2Cr_2O_7$ titration of uranium(IV), mg U/g $K_2Cr_2O_7$ solution,
 C_c = concentration of $K_2Cr_2O_7$ solution from 11.2.2, mg $K_2Cr_2O_7$ /g $K_2Cr_2O_7$ solution, and
 M = equivalent multiplier for $K_2Cr_2O_7$ to uranium conversion defined for the titration reaction and the atomic weight of the standard uranium, no units (2.42734 for CRM 112-A or its equivalent).

11.3.3 When a $K_2Cr_2O_7$ solution is standardized against a standard uranium solution, the titration factor (mg uranium/g $K_2Cr_2O_7$ solution) is calculated directly from the standardization titration using the following equation:

$$T = (C_u)(G)/(W) \quad (7)$$

where:

T = titrant factor for $K_2Cr_2O_7$ titration of uranium(IV), mg uranium/g $K_2Cr_2O_7$ solution,
 C_u = concentration of standard uranium solution from 11.2.3, mg uranium/g uranium solution,
 G = weight of standard uranium solution in the aliquant of CRM 112-A uranium metal or its equivalent, g uranium solution, and
 W = weight of $K_2Cr_2O_7$ solution used as titrant, g $K_2Cr_2O_7$ solution.

11.4 The weight of uranium calculated for samples using the uranium titration factor calculated in 11.3 must be corrected for atomic weight differences between the sample and CRM 112-A or its equivalent.

11.4.1 *Sample Result*—Calculate the uranium content of the original sample by the following equation:

$$U = TWR/FS \quad (8)$$

TABLE 1 Summary of % Relative Difference Data from Titration of a Uranyl Nitrate^A Solution by Three Laboratories Over Four Years Using the Modified Davies and Gray Method^B

Lab	<i>n</i> ^C	Mean		Standard Deviation	
A	382	−0.039		0.033	
B	168	−0.044		0.052	
C	66	−0.046		0.072	
C	64 ^D	−0.055		0.055	
Summary	<i>n</i>	Within Lab		Due to Lab	
		Mean	Variation	Variation	Repro. ^E
Pooled Results	616	−0.041	0.044	0.002	0.044
Pooled Results	614 ^D	−0.042	0.042	0.005	0.042
Labs A and B ^F	550	−0.041	0.040	0.002	0.040

^A The 95 % confidence interval of the mean determined for the reference solution uranium concentration is 0.6817 ± 0.00073 % uranium.

^B All data is available from ASTM, which includes dates of analysis, number of analysts, and duplicate sample indications.

^C *n* is the total number of titrations, some of which were performed on the same day by the same analyst and some of which were performed at a different time, or by a different analyst, or both.

^D Excluding two statistical outliers which may have been due to previous lack of attention to procedural details that are now identified within this procedure.

^E Reproducibility is the variation due to all factors including between laboratory variations. It is equal to:

$$\sqrt{(\text{Within Lab Variation})^2 + (\text{Variation Due to Lab})^2}$$

^F Laboratories A and B are grouped separately because Laboratory C was statistically significantly different from Laboratories A and B, and Laboratory C had statistical outliers.

where:

- U* = uranium per g sample, mg U/g sample,
- T* = titration factor, mg uranium/g K₂Cr₂O₇ solution, calculated in 11.3.2 or 11.3.3,
- W* = weight of K₂Cr₂O₇ solution, g K₂Cr₂O₇ solution,
- R* = ratio of atomic weight of uranium in sample to atomic weight of CRM 112-A or its equivalent,
- F* = factor for sample dilution, g sample per g sample solution, and
- S* = weight of sample solution aliquant analyzed, g.

12. Precision and Bias⁹

12.1 The uranium titration factor (11.3), and so the calibration of the method, is based on CRM 112-A, uranium reference material or its equivalent, or on SRM 136e, K₂Cr₂O₇ reference material or its equivalent.

12.2 This test method has been used for titration of uranium from product, fuel, and scrap material (1) after dissolution and treatment. The precision and bias information for the uranium titration, contained in this section, was derived from a uranyl nitrate solution titration by three laboratories. No recent interlaboratory data is available for any other material type. The International Target Values (Precision = 0.15 % and Bias = 0.15 %) (20) for the titration of pure uranium solutions is approximately three times that determined by this study. The International Target Values for the titration of other material types have precisions (% RSD) ranging from 0.15 to 0.5 % and biases ranging from 0.1 to 2.0 % depending upon the purity of the material. Uncertainties associated with sample assay other than those for the uranium titration, that is, due to sampling, sample handling, and sample weight determination are in-

cluded neither in the precision and bias information for this test method nor in the International Target Values. Uncertainties for sampling, sample handling, and sample weight determination should be part of the calculation of the total uncertainty for determining uranium content by titration or any other assay method.

12.3 An interlaboratory program, the Safeguards Measurement Evaluation Program, in which the NBL provides materials to participating facilities for analysis was used to provide both interlaboratory and intralaboratory comparisons of analysis of the same material. Data from these analyses on uranyl nitrate solutions, prepared by the NBL and distributed to three laboratories for titration by the Modified Davies and Gray Method, were used as the basis for the statistical evaluation of the test method.¹⁰ The data is summarized in Table 1 for three laboratories A, B, and C.

12.4 The test method, as applied to uranyl nitrate solutions, shows no statistically significant bias. The data in Table 1 (pooled results) give an estimated relative bias of −0.042 % which is well within the stated uncertainty of the material (see footnote ^A in Table 1). The variation due to the laboratory factor for the analysis technique is not statistically significant (0.005 % of the reference value of 0.6817 % uranium). The within laboratory variation (% RSD) of the analysis technique is 0.042 % of the reference value.

13. Keywords

13.1 chromium titration of uranium; Davies and Gray titration of uranium; Modified Davies and Gray titration of uranium; uranium analysis; uranium analysis in fuel; uranium analysis in product

⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C26-1004.

¹⁰ Safeguard Measurement Evaluation Program Data, New Brunswick Laboratory, 1987–1990, private communication.

ANNEXES

(Mandatory Information)

A1. INTERFERENCES IN THE URANIUM TITRATION WITH TREATMENT STRATEGY

See [Table A1.1](#).

TABLE A1.1 Elemental Interference in the Modified Davies and Gray Uranium Titration with Treatment Strategy

Element/s	Treatment Strategy
Al	Hydrochloric and nitric acid dissolution. Filter residue and ignite at 900°C; combine with solution, and fume with 1 + 1 sulfuric acid (2).
Zr-Mo	Fume sample in 1 + 1 sulfuric, concentrated nitric and hydrofluoric acids (2).
V, Bi	Reduce the sample size (2).
Zr	Add 1 to 2 mL of hydrofluoric acid to the sample before titrating (2).
As, Sb, Sn	Add potassium dichromate to sample prior to reduction step (8). ^A
Ru, Os	Fume sample three times with perchloric and nitric acids (12).
Mo, Cl, Br, F	Fume sample with sulfuric and/or nitric and perchloric acids (2, 12, 13).
I	Add bromine water to sample, evaporate, fume with sulfuric or nitric and perchloric acid (8).
Tc	Fume sample with sulfuric or nitric and perchloric acid to dryness; flame walls of beaker to insure dryness (14).
Ag, Hg, Pt, Pd	Use a copper column separation (8, 15).
>10 % Au	Reduce the gold to metal and separate (8).
Am, Sb, As, Br, Cl, Fe, Au, Pb, Ir, Mn, Hg, Mo, Np, Os, Pd, Pt, Pu, Ru, Ag, Th, Sn, V, Zr	Use the tributyl phosphate/carbon tetrachloride extraction technique (16). Alternate solvents such as dodecane are now used.

^A The potassium dichromate oxidizes these elements to a non-interfering oxidation state which the ferrous ion is not capable of reducing.

A2. TREATMENT OF PHOSPHORIC ACID TO OXIDIZE REDUCING SUBSTANCES

A2.1 Add 1 mL of 2 % potassium dichromate solution (prepared in 7.8) to a 2.4 L (5 pint) bottle of phosphoric acid for oxidation of reducing impurities. If the resulting bottle of phosphoric acid does not maintain a light yellow straw color over 2 to 3 h, add an additional 1 mL of 2 % dichromate and allow the solution to sit 8 h. If the solution maintains the straw color, the phosphoric is acceptable for use. If the solution is light green in color, do not use the phosphoric acid or any other phosphoric acid bottles with the same preparation lot number (8, 21-23).

A2.2 Alternatively, add 2 drops of 2 % potassium dichromate solution (prepared in 7.8) to 40 mL of phosphoric acid prior to addition to the sample for titration. It is suggested that, prior to using a new preparation lot of phosphoric acid, one bottle be tested either by the ACS test for reducing substances in phosphoric acid or by the test in A2.1.

APPENDIXES

(Nonmandatory Information)

X1. POLYETHYLENE BOTTLE WEIGHT BURET PREPARATION

X1.1 A 125-mL polyethylene bottle with a detachable tip drawn by heating low density polyethylene tubing, $\frac{5}{32}$ in. inside diameter (I.D.) and $\frac{1}{4}$ in. outside diameter (O.D.), and pulling to a fine tip which is attachable to the bottle's own normal tip for the final end point approach in the weight Modified Davies and Gray titration of uranium (24) has been

found to allow dichromate additions by weight with good precision and accuracy. A tip drawn small enough to deliver about 5 mg of dichromate solution per drop has been found to allow acceptable rates of dichromate addition near the end point.

X2. PLATINUM WIRE ELECTRODE TREATMENTS

X2.1 A routine platinum wire electrode cleaning treatment and the treatment for restoring fast response to a sluggish platinum electrode is flaming to white heat with immediate immersion in concentrated nitric acid; the process is usually done at least two times.

X2.2 An alternate cleaning method for the platinum wire electrodes is to soak the electrode in hydrofluoric acid prior to

flaming and nitric acid immersion as detailed in X2.1.

X2.3 If the analyses of standards show increasing bias over the day's analyses after routine cleaning, for several consecutive days, a sodium bisulfate fusion of the platinum wire electrode may be needed. After the sodium bisulfate fusion and flaming, again perform the routine cleaning in X2.1.

X3. DICHROMATE SOLUTION STANDARDIZATION

X3.1 Standardize reagent grade or better potassium dichromate against CRM 112-A uranium metal or its equivalent (see Appendix X4).

X3.2 The standard deviation of the mean for the potassium dichromate standardization (s/\sqrt{n}) should be well within the accepted laboratory error limits for the uranium titration when

$n = 10$. Example: To maintain an accepted laboratory precision on the uranium titration of $s = 0.10$, the recommended acceptable precision on the potassium dichromate standardization is $s = 0.05$ for $n = 10$ so that the accuracy of the potassium dichromate standard solution will have a minimal effect on the titration results.

X4. DISSOLUTION OF URANIUM METAL

X4.1 The following method has been used successfully for the dissolution of approximately 40 g of uranium metal for which the cleaned and dried weight of the uranium metal has been accurately determined. The surface of the uranium metal (CRM 112-A or its equivalent) shall be cleaned following the instructions on the certificate.

X4.2 Place the metal in a dry weighed 2-L flask, or suitable alternative, and add 100 mL of 8 M nitric acid.

X4.2.1 Place a small funnel, sitting on a glass bend, in the mouth of the flask, and place the flask on a hot plate at about 75°C until dark brown fumes are seen.

X4.2.2 Lower the hot plate temperature to about 65°C, and heat for 6 h. Swirl the mixture occasionally during this stage of dissolution.

X4.3 Remove the flask from the hot plate, and add 50 mL of 8 M nitric acid.

X4.4 Return the mixture to the hot plate with the funnel in the mouth of the flask, turn the temperature of the hot plate to about 75°C, and leave the mixture for 8 h.

X4.5 Add 50 mL of 8 M nitric acid to the flask, turn the hot plate to about 85°C, leave the mixture on the hot plate for 4 h with occasional swirling. Visually check for completeness of dissolution.

X4.5.1 If the dissolution is complete, dilute to 2 L with water, and allow the solution to equilibrate to room temperature.

X4.5.2 Otherwise, continue heating the mixture until dissolution is complete.

X4.6 If insufficient nitric acid was available for dissolution, the surface of the metal may become passive, and complete dissolution will not be possible; in that case, the mixture should be discarded.

X4.7 Weigh the equilibrated solution, make buoyancy and purity corrections to the uranium metal weight, and calculate the concentration of the uranium solution.

X4.8 Thoroughly mix the uranium solution, separate into multiple borosilicate bottles with polycone seals for long-term storage, or aliquant into beakers covered with petri dishes or a suitable alternative for storage.

X4.9 Aliquants may be stored after drying as the nitrate or fumed in sulfuric acid and stored as the sulfate.

X4.10 A suitable time period for dissolution of stored dried aliquants before titration must be used; 30 min to 1 h is suggested. Dried aliquants which are not allowed adequate dissolution time may give low titration results.

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