Designation: C761 − 11

Standard Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride[1]

This standard is issued under the fixed designation C761; 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 These test methods cover or give reference to procedures for subsampling and for chemical, mass spectrometric, spectrochemical, nuclear, and radiochemical analysis of uranium hexafluoride (UF_6). Most of these test methods are in routine use to determine conformance to UF_6 specifications in the Enrichment and Conversion Facilities.

1.2 The analytical procedures in this document appear in the following order:

NOTE 1—Subcommittee C26.05 will confer with C26.02 concerning the renumbered section in Test Methods C761 to determine how concerns with renumbering these sections are best addressed in subsequent publications as analytical methods are replaced with stand-alone analytical methods.

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 consideration statements, see Section 6.)

2. Referenced Documents

2.1 The following documents of the issue in effect on date of material procurement form a part of this specification to the extent referenced herein:

2.2 *ASTM Standards:*[2]

C787 Specification for Uranium Hexafluoride for Enrichment

C799 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions

C859 Terminology Relating to Nuclear Materials

C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U

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

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[1] These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.05 on Methods of Test.

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[2] 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.

C1219 Test Methods for Arsenic in Uranium Hexafluoride (Withdrawn 2015)[3]
C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
C1267 Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium
C1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry
C1295 Test Method for Gamma Energy Emission from Fission and Decay Products in Uranium Hexafluoride and Uranyl Nitrate Solution
C1344 Test Method for Isotopic Analysis of Uranium Hexafluoride by Single-Standard Gas Source Mass Spectrometer Method
C1346 Practice for Dissolution of UF_6 from P-10 Tubes
C1380 Test Method for the Determination of Uranium Content and Isotopic Composition by Isotope Dilution Mass Spectrometry
C1413 Test Method for Isotopic Analysis of Hydrolyzed Uranium Hexafluoride and Uranyl Nitrate Solutions by Thermal Ionization Mass Spectrometry
C1428 Test Method for Isotopic Analysis of Uranium Hexafluoride by Single–Standard Gas Source Multiple Collector Mass Spectrometer Method
C1429 Test Method for Isotopic Analysis of Uranium Hexafluoride by Double-Standard Multi-Collector Gas Mass Spectrometer
C1441 Test Method for The Analysis of Refrigerant 114, Plus Other Carbon-Containing and Fluorine-Containing Compounds in Uranium Hexafluoride via Fourier-Transform Infrared (FTIR) Spectroscopy
C1474 Test Method for Analysis of Isotopic Composition of Uranium in Nuclear-Grade Fuel Material by Quadrupole Inductively Coupled Plasma-Mass Spectrometry
C1477 Test Method for Isotopic Abundance Analysis of Uranium Hexafluoride and Uranyl Nitrate Solutions by Multi-Collector, Inductively Coupled Plasma-Mass Spectrometry
C1508 Test Method for Determination of Bromine and Chlorine in UF_6 and Uranyl Nitrate by X-Ray Fluorescence (XRF) Spectroscopy
C1539 Test Method for Determination of Technetium-99 in Uranium Hexafluoride by Liquid Scintillation Counting
C1561 Guide for Determination of Plutonium and Neptunium in Uranium Hexafluoride and U-Rich Matrix by Alpha Spectrometry
C1636 Guide for the Determination of Uranium-232 in Uranium Hexafluoride
C1689 Practice for Subsampling of Uranium Hexafluoride
C1742 Test Method for Isotopic Analysis of Uranium Hexafluoride by Double Standard Single-Collector Gas Mass Spectrometer Method
D1193 Specification for Reagent Water
D3084 Practice for Alpha-Particle Spectrometry of Water
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

2.3 *American Chemical Society Specification:*

Reagent Chemicals [4]

2.4 *Other Specifications:*

Uranium Hexafluoride : Base Charges, Use Charges, Special Charges, Table of Enriching Services, Specifications, and Packaging[5]
USEC 651 Good Handling and Practices for UF_6

2.5 *ANSI Standards:*[6]

ANSI N 14.1 Nuclear Material-Uranium Hexafluoride-Packaging for Transport

2.6 *ISO Standards:*

ISO 7195 Nuclear Energy-Packaging of Uranium Hexafluoride (UF_6) for Transport

3. Significance and Use

3.1 Uranium hexafluoride is a basic material used to prepare nuclear reactor fuel. To be suitable for this purpose the material must meet criteria for uranium content, isotopic composition, metallic impurities, hydrocarbon and halohydrocarbon content. These test methods are designed to determine whether the material meets the requirements described in Specifications C787 and C996.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all procedures. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided that it is first established that the reagent to be used is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean reagent water conforming to Specification D1193.[7]

5. Rejection

5.1 Rejection or acceptance criteria are described in Specifications C787 and C996.

6. Safety Considerations

6.1 Since UF_6 is radioactive, toxic, and highly reactive, especially with reducing substances and moisture (see Uranium Hexafluoride: Handling Procedures and Container Criteria, sections 2.4 through 2.6), appropriate facilities and practices for sampling and analysis must be provided.

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[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] *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.

[5] United States Department of Energy, Oak Ridge, TN 37830.

[6] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

[7] Type 1 and 2 water have been found to be suitable.

6.2 Hydrofluoric acid is a 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 the burn depends on the concentration, the temperature, and the duration of the contact with the acid. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep issue 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 personal protective equipment to protect from skin and eye contact is essential.

6.3 *Committee C-26 Safeguards Statement:*

6.3.1 The material (uranium hexafluoride) to which these test methods apply, is subject to nuclear safeguards regulations governing its possession and use. The following analytical procedures in these test methods have been designated as technically acceptable for generating safeguards accountability measurement data: Gravimetric Determination of Uranium; Titrimetric Determination of Uranium; All Isotopic Analyses.

6.3.2 When used in conjunction with appropriate certified Reference Materials (CRMs), these procedures can demonstrate traceability to the national measurement base. However, adherence to these procedures does not automatically guarantee regulatory acceptance of the resulting safeguards measurements. It remains the sole responsibility of the user of these test methods to assure that its application to safeguards has the approval of the proper regulatory authorities.

SUBSAMPLING OF URANIUM HEXAFLUORIDE

7. Scope

7.1 This test method has been discontinued (see C 761–04$^{\varepsilon 1}$). The subsampling of UF$_6$ from bulk sample containers into smaller containers suitable for laboratory analyses has been published as a separate Practice, C1689.

GRAVIMETRIC DETERMINATION OF URANIUM

8. Scope

8.1 Practice C1346 is applicable to the hydrolysis of uranium hexafluoride in polychlorotrifluoroethylene (P10) tubes. The following test method is then applicable to the direct gravimetric determination of uranium.

9. Summary of Test Method

9.1 A sample of uranium hexafluoride is weighed, cooled in liquid nitrogen, and hydrolyzed with water. The uranyl fluoride solution produced is evaporated to dryness and converted to uranic oxide by pyrohydrolysis. The uranium content is determined from the weight of the uranium oxide after correcting for stoichiometry based on isotopic content, ignition conditions, and nonvolatile impurities. Ref. (**1-4**).[8]

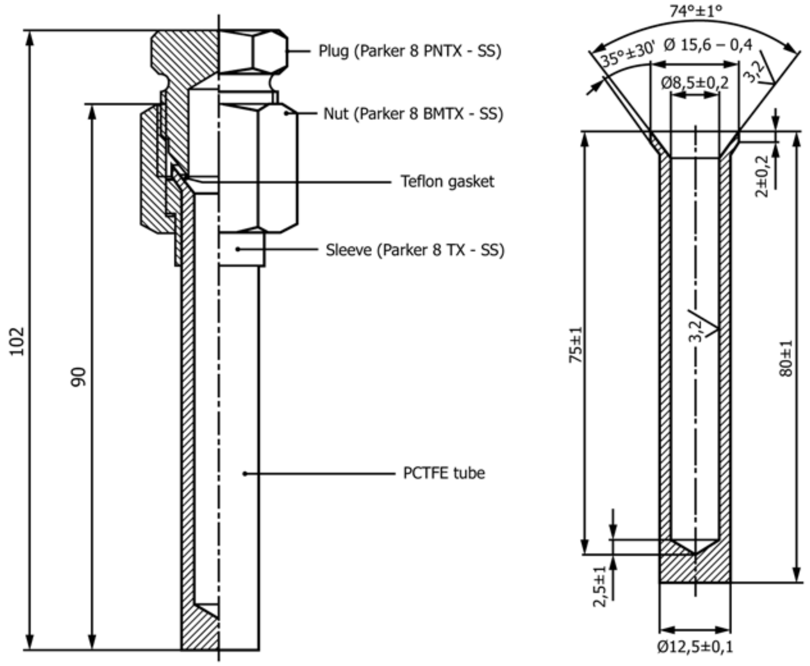

FIG. 1 Example of a Polychlorotrifluoroethylene P-10 Tube

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[8] The boldface numbers in parentheses refer to a list of references at the end of these test methods.

10. Interferences

10.1 Nonvolatile impurities affect the accuracy of the method and must be measured by spectrographic analysis with corrections applied.

11. Apparatus

11.1 *Polytrifluorochloroethylene (PTFCE) Sample Tube, TFCE Gasket, Flare Nut, and Plug,* see Fig. 1.

11.2 *Platinum Boat and Cover*—The cover should be platinum gauze (52 mesh) and shaped to cover the boat (Fig. 2).

11.3 *Muffle Furnace,* must be capable of operating continuously at 875°C and maintain this temperature within ±25°C. The furnace shall be equipped with a steam supply that is passed through a tube furnace to preheat the steam to 875°C.

11.4 *Tube Furnace,* must be capable of operating continuously at 875°C and maintain this temperature within 25°C.

11.5 *Infrared Heat Lamps,* 250 watts.

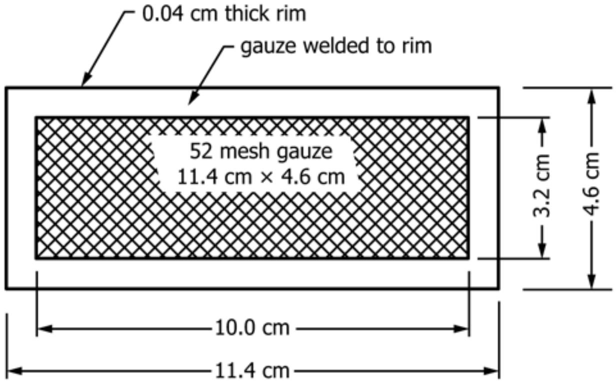
11.6 *Analytical Balance.*

11.7 *Vacuum Oven.*

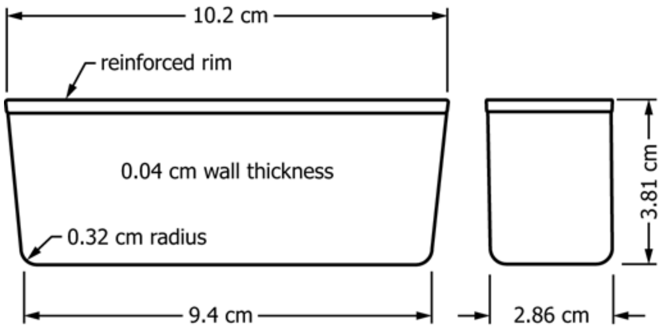
11.8 *Dewar Flask,* stainless steel.

11.9 *Spatula,* platinum.

11.10 *PTFCE Rod,* 120 mm long and 1.6 mm in diameter.



Cover



Boat

FIG. 2 Platinum Boat and Cover

11.11 *Forceps,* platinum tipped.

11.12 *Jig,* suitable for holding the TFCE sample tube so that it can be opened with a wrench.

11.13 *Box Wrench,* to fit sample tube plug.

11.14 *Beaker,* stainless steel, 125 mL capacity.

12. Reagents

12.1 *Liquid Nitrogen.*

12.2 *Nitric Acid (sp gr 1.42)*—concentrated nitric acid (HNO_3).

12.3 *Nitric Acid* (4*M*)—Mix 500 mL of concentrated HNO_3 with 1500 mL of distilled water.

12.4 *Detergent.*

13. Sampling

13.1 A UF_6 sample is taken as described in Practice C1689.

14. Procedure

14.1 Inspect the PTFCE sample tube for leaks.

NOTE 2—An indication of a leak is a yellow-green residue on the flare nut and cap or a yellow discoloration in the tube. Discard the sample if a leak is indicated.

14.2 Allow the sample tube to stand overnight in the laboratory.

14.3 Wipe the sample tube with a lint-free tissue to remove any moisture or foreign material that might be adhering.

14.4 Weigh the sample tube to the nearest 0.1 mg.

14.5 Heat the platinum boat and screen in the pyrohydrolysis furnace at 875°C for 20 min.

14.6 Cool the platinum boat and store in a desiccator for 40 min. Weigh the boat and screen to the nearest 0.1 mg.

14.7 Freeze the sample by immersing the sample tube in liquid nitrogen for 10 min.

14.8 Add enough chilled water to the tared platinum boat to immerse the sample tube (about 50 mL).

14.9 Place the sample tube in the jig and loosen the plug with the box wrench.

14.10 Remove the sample tube from the jig and unscrew the plug while holding the sample tube in an upright position.

14.11 Remove the flare nut from the sample tube and immerse the tube and gasket in the chilled water in the tared platinum boat.

14.12 Let the gasket remain in the chilled water about 30 min.

14.13 Remove the gasket with the forceps and rinse well with deionized water into the boat.

14.14 Place the plug-nut assembly and gasket into a stainless steel beaker for drying.

14.15 Allow the tube to remain in the water until the UF_6 has been hydrolyzed (2 to 4 h).

14.16 Remove the tube from the sample solution by inserting the TFCE rod or platinum spatula into the tube and lifting directly above the boat.

14.17 Rinse the sample tube with deionized water into the boat using extreme care to prevent splashing.

14.18 Cover the sample boat containing the UO_2F_2 solution with the matching cover shown in Fig. 2. Place under the infrared head lamps and evaporate to dryness for 16 h.

14.19 Shake the excess water from the sample tube and place in the stainless beaker containing the plug-nut assembly and gasket.

14.20 Dry the sample tube parts in the vacuum oven at 80°C.

14.21 Allow the unassembled parts to sit in the room overnight.

14.22 Assemble the empty sample tube and weigh to the nearest 0.1 mg.

14.23 Disassemble the sample tube and soak the tube and gasket in 4*M* HNO_3 at 75° to 80°C for 1 h.

14.24 Rinse with deionized water and place in the stainless steel beaker.

14.25 Clean the metal parts with detergent and rinse with deionized water and acetone.

14.26 Place the metal parts to the stainless steel beaker and dry all parts in the vacuum oven at 80°C overnight.

14.27 Reassemble the sample tube for the next sample.

14.28 Set the temperatures of the furnace and tube furnace at 875°C.

14.29 Establish a steam flow to the furnace equal to 1 L of water per hour.

14.30 Place the boat into the furnace with the platinum cover on the boat and pyrohydrolyze the sample for 1 h.

14.31 Remove the boat from the furnace, cool, and place in a desiccator while still warm.

14.32 Desiccate the sample for 1 h and weigh quickly to the nearest 0.1 mg.

14.33 Transfer a portion of the U_3O_8 residue to a vial and submit for spectrographic analysis to determine the weight of nonvolatile impurities.

14.34 Place the platinum boat in hot 4*M* HNO_3 for 3 to 4 h and rinse with deionized water acetone.

15. Calculation

15.1 Calculate the weight fraction of uranium in the sample as follows:

$$\text{gU/g UF}_6 = (A - (AB))(\text{Gravimetric Factor})/W \tag{1}$$

where:

Gravimetric Factor = gU/g U_3O_8 which varies with isotopic composition. Theoretical stoichiomtry for U_3O_8 cannot be assumed and the actual gU/g U_3O_8 must be established by potentiometric titration **(1–4)**. (Tri-diffusion plant committee with DOE approval has established 0.8479 g U/g U_3O_8 by titration as the factor for natural uranium,

A = grams of U_3O_8 from the pyrohydrolysis of UO_2F_2,

B = grams of impurity metal oxides per gram of U_3O_8,

W = corrected sample weight in grams.

The correction is for the combined effects of cover gas trapped over the UF_6 in the sample tube and the air buoyancy correction **(5)**. The following equation has been determined for the sample tube in Fig. 1 and the subsampling conditions described in Practice C1689. The correction equation is applicable for sample weights in the range of 7 to 13 g.

$$W = (1.00047)x - 0.0058 \tag{2}$$

where:

x = observed UF_6 sample weight, g.

16. Precision and Bias

16.1 *Precision*—The precision within a laboratory and between laboratories was established by analyzing 15 samples at each laboratory. The sampling scheme is shown in Table 1. Within a laboratory, based on 15 measurements made on separate days the relative standard deviation is 0.021 %. The results from all the laboratories are shown in Table 2.

16.2 *Bias*—To establish an estimate of bias for the gravimetric method, a series of comparative analyses of UF_6 control batches were made using the gravimetric and potentiometric titration methods. The potentiometric titration was used as the reference method because the uranium was measured directly using NIST potassium dichromate.[9] The results are shown in Table 3.

TITRIMETRIC DETERMINATION OF URANIUM

17. Scope

17.1 A sample of the U_3O_8 produced by the hydrolysis of the UF_6 and ignition of the resulting UO_2F_2 is analyzed according to Test Method C1267.

PREPARATION OF HIGH-PURITY U_3O_8

18. Scope

18.1 High purity U_3O_8 can be prepared according to Preparation C1128. High purity uranium is needed for a blank matrix for analyses using ICP-MS, ICP-AES, AA, XRF, and MS equipment.

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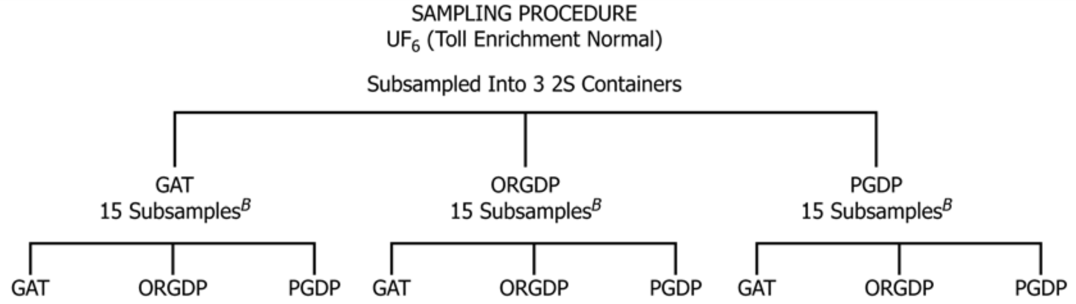
[9] Standard reference material, now available as NIST SRM 136e.

TABLE 1 Interlaboratory Study[A]—Determination of Uranium in Uranium Hexafluoride



[A] Date of study—October 1983; Participating laboratories—Goodyear Atomic Corporation, Portsmouth, Ohio (GAT); Martin Marietta Energy Systems, Inc., Oak Ridge, Tennessee (ORGDP); Martin Marietta Energy Systems, Inc., Paducah, Kentucky (PGDP).

[B] Five tubes analyzed at each laboratory.

TABLE 2 Results of Interlaboratory Study—U in UF_6

Analysis Site %U in UF_6		
GAT	ORGDP	PGDP
Subsampled at GAT:		
67.600	67.619	67.589
67.601	67.574	67.575
67.583	67.607	67.612
67.611	67.600	67.612
67.618	67.606	Sample Lost
Subsampled at ORGDP:		
67.614	67.580	67.611
67.611	67.621	67.598
67.587	67.600	67.501
67.599	67.606	67.610
67.617	67.596	67.624
Subsampled at PGDP:		
67.616	67.588	67.591
67.586	67.602	67.620
67.573	67.612	67.612
67.614	67.606	67.612
67.607		67.586
Mean and Standard Deviation:		
67.602 ± 0.014	67.601 ± 0.013	67.603 ± 0.014

ISOTOPIC ANALYSIS

19. Scope

19.1 The isotopic composition can be determined on either gaseous UF_6 or on hydrolyzed UF_6.

19.2 For gaseous UF_6, using single collector mass spectrometer instruments, Test Methods C1344 and C1742 have been developed and can be used for single or double standard method respectively. For multi-collector instruments, Test Methods C1428 and C1429, using single or double standard can be used.

19.3 For hydrolyzed UF_6, methods using Thermal Ionization Mass Spectrometry (TIMS) have been developed and can be used: Test Methods C1413 and C1380. Methods using ICP-MS can also be used: Test Methods C1474 and C1477.

DETERMINATION OF HYDROCARBONS, CHLOROCARBONS, AND HALOHYDROCARBONS

20. Scope

20.1 The determination of some forms of hydrocarbons, chlorocarbons, and halohydrocarbons in UF_6 vapor can be performed using Test Method C1441. As an alternative, a mass spectrometry technique may be used and is detailed below. Although this test method is only semiquantitative, it is adequate for certifying that the subject impurities do not exceed 0.01 mol % of the UF_6.

21. Summary of Test Method

21.1 UF_6 is admitted to a mass spectrometer through a gas sample leak, and magnetic scanning is employed to record a spectrum of peaks. A representative group of recorded peaks is compared to the same peaks in a pure UF_6 standard scan to determine whether appreciable ion fragments from subject impurities are present.

22. Interferences

22.1 If detectable impurities are present, a complete mass scan of the range from 12 to 400 is performed. All impurities are then identified from their cracking patterns, and calculations are performed using ionization efficiency factors for the compounds present. Since cracking patterns vary with ionization potential and ionization efficiencies vary with focus conditions, this measurement can only be performed by one proficient in analytical mass spectrometery.

23. Apparatus

23.1 A mass spectrometer with resolution adequate to distinguish between adjacent peaks at $m/e = 400$ is required. For example, a 152-mm radius, 60-deg, Nier-type spectrometer modified for spectrum recording **(6)** is suitable. The sample inlet system should be of nickel or Monel, equipped with an

TABLE 3 Determination of Uranium in Uranium Hexafluoride—Comparison of Gravimetric and Potentiometric Titration Methods

Control UF_6 Date[A]	Method	Number of Measurements	%Uranium		Bias Estimate[B]
			Mean	SD	
9/78	Gravimetric	24	67.610	0.009	−0.001
	Potentiometric Titration	9	67.611	0.015	
5/82–8/82	Gravimetric	30	67.596	0.010	−0.009
	Potentiometric Titration	8	67.605	0.011	
7/83–9/83	Gravimetric	25	67.610	0.006	+ 0.005
	Potentiometric Titration	8	67.605	0.010	

[A] Control UF_6 used in 9/78 was a different batch of material from that used in 1982 and 1983.

[B] Potentiometric titration results are used as the reference values for the bias estimates.

adjustable viscous-flow or molecular leak for delivering the sample to the ion source.

23.2 The ion source must be fabricated from nonmagnetic material such as Nichrome V, and must be designed so it can be disassembled for cleaning. The magnetic field of the analyzer magnet must be continuously variable from about 200 to 6500 gauss. A single ion collector electrode is suitable, and a vibrating-reed electrometer and 304-mm strip chart recorder are optimum for amplifying and recording ion signals.

23.3 It is quite possible that quadrupole or time-of-flight instruments could be adapted to this measurement.

24. Procedure

24.1 *UF_6 Standard Measurements:*

24.1.1 Select a standard material that has been given repetitive flash purifications to rid it of all volatile impurities. Isotopic UF_6 standards usually fall in this category.

24.1.2 With the electrometer sensitivity set at 1⁄100 of the most sensitive usable operating range, adjust the gas flow to the ion source to record a mass (Note 3) 333 peak (UF_5^+) approximately 80 % of full scale (80 divisions).

NOTE 3—The term “mass” in this procedure alludes to *m/e*, the mass-to-charge ratio (see also Terminology C859).

24.1.3 Measure the ratio of mass 333 (UF_5^+) to 147.5 (UF_3^{++}). Mass 333 is measured on a sensitivity range one-hundredth that of mass 147.5. Depending on focus conditions, a ratio of the order of 10^2 is obtained. Measure this ratio only once per day and use for calculating results of all samples analyzed that day.

24.1.4 With the electrometer sensitivity set at 1⁄100 of the most sensitive usable operating range, increase the gas flow to provide an output signal of approximately 80 divisions at the 147.5 mass position. This gives a detection limit of the order of 2 ppm per chart division: 50 to 100 due to ratio between UF_5^+ and UF_3^{++}, 100 due to sensitivity shunts, and approximately 80 on the recorder chart.

24.1.5 Record a scan of mass range from 12 to 150 using the most sensitive usable operating shunt, and use this scan as a background for all samples analyzed that day.

24.2 *UF_6 Sample Measurement:*

24.2.1 Introduce the sample to the spectrometer source such that an output intensity of approximately 80 chart divisions is obtained at the 147.5 mass number (UF_3^{++}), using 1⁄100 the most sensitive usable operating range.

24.2.2 Record a scan of mass range from 12 to 150 using the most sensitive usable operating range.

24.2.3 Repeat 24.2.1 and 24.2.2 for each sample to be analyzed that day.

25. Calculation

25.1 Due to mass spectrometer cracking patterns, low-mass ion fragments are produced from all compounds, even the high-mass ones.

25.2 It is practical to look for a representative group of such ion fragments at specific masses. Thus, initially monitor the following masses for purposes of this procedure:

Mass Number	Positively Charged Ion Fragment
15	CH_3
26	C_2H_2
27	C_2H_3
31	CF
43	C_3H_7
47	CCl^{35}
49	CCl^{37}
69	CF_3

25.3 Read sample intensities for the representative ion fragments from the recorder chart.

25.4 Subtract the background intensities observed on the pure standard from respective sample intensities.

25.5 Examine the net intensity at each of the eight mass numbers. (It will be recalled that one recorder chart division of net intensity is equivalent to about 2 ppm on a UF_6 basis; however, ionization efficiencies of compounds differ, and a specific ion fragment may result from many different compounds. Thus, the net intensity at a specific mass number is only qualitative and not a quantitative measurement of impurity.)

25.5.1 If the net ion intensity does not exceed 2 ppm at any of the mass positions, report the sample as containing less than 0.01 mol % of the subject impurities.

25.5.2 Where detectable impurities are apparent, perform a complete mass scan of the range from 12 to 400, identify impurities, and perform calculations using ionization efficiency factors for the compounds present.

26. Reliability

26.1 This simplified procedure was designed specifically to certify that a UF_6 sample contains less than 0.01 mol %

hydrocarbons, chlorocarbons, and partially substituted halohydrocarbons. Thus, the procedure is qualitative rather than quantitative in cases where the impurity level is below 100 ppm.

26.2 The detectability limit for any ion fragment is about 2 ppm. The detectability limit for the parent compound could be greater or less than 2 ppm depending on ionization efficiency and cracking pattern. If one of the subject compounds were present to 10 ppm or more, it would be evident from monitoring the eight masses. Thus, a quoted result of less than 0.01 mol % is conservative.

26.3 The results are considered quantitative when the impurity being determined is present to a level greater than 100 ppm. In such instances the impurity is identified and measured. The 95 % symmetrical confidence interval for such a measurement is ±50 % of the quoted impurity.

DETERMINATION OF ANTIMONY

27. Scope

27.1 The Atomic Absorption test method has been discontinued (see C761–96). Antimony can be determined by ICP-MS. Test Method C1287 can be used.

DETERMINATION OF BROMINE

28. Scope

28.1 The Spectrophotometric test method has been discontinued (see C761–96). Bromine can be determined by X-Ray spectroscopy. Test Method C1508 can be used.

DETERMINATION OF CHLORINE

29. Scope

29.1 Chlorine can be determined by X-Ray Spectroscopy. Test Method C1508 can be used. Chlorine can also be determined by titrimetry. This test method is described below. It is applicable over a range from 10 to 100 ppm chlorine; however, higher concentrations can be measured by appropriate sample dilution.

30. Summary of Test Method

30.1 The test method consists of treating a hydrolyzed sample of UF_6 with ferrous sulfate in sulfuric acid solution to reduce chlorates, and then with potassium permanganate to liberate free chlorine. The chlorine gas is carried by a nitrogen stream into a potassium iodide solution, and the liberated iodine is titrated with sodium thiosulfate. Bromine, if present, is determined separately, and a correction is applied to the chlorine result.

30.2 It is recommended that the potassium iodide-sodium acetate solution be made up fresh once each week. Any color change signals the need for a new solution. As the solution ages, the blank result increases; therefore, the same potassium iodide solution is used for both sample and blank.

30.3 If the sample solution is allowed to boil too vigorously when chlorine gas is being released, liquid droplets may be carried by the nitrogen stream into the potassium iodide solution, resulting in sample bias.

31. Apparatus

31.1 *Distillation Apparatus,* shown in Fig. 3.

32. Reagents

32.1 *Boric Acid* (H_3BO_3), reagent grade, crystal or powder.

32.2 *Ferrous Sulfate Solution*—5 g $FeSO_4 \times 7H_2O$ dissolved in 500 mL of 3.6 *M* sulfuric acid.

32.3 *Potassium Iodide* (KI), reagent grade.

32.4 *Potassium Iodide-Sodium Acetate Solution*—Dissolve 100 g of KI and 100 g of $NaC_2H_3O_2 \times 3H_2O$ in distilled water and dilute to 2 L.

32.5 *Potassium Permanganate Solution (1 %)*—Prepare a 1 % solution of $KMnO_4$ in water.

32.6 *Sodium Acetate* ($NaC_2H_3O_2$), reagent grade.

32.7 *Sodium Thiosulfate Solution* (0.025 *N*)—Prepare a 0.025 *N* solution of $Na_2S_2O_3$ in water.

32.8 *Starch Indicator Solution,* pH 7.

32.9 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4).

33. Procedure

33.1 *Sample Preparation:*

33.1.1 Hydrolyze the sample of UF_6 with distilled water. Approximately 250 g of UF_6 from a nickel knockout container may be hydrolyzed to provide a sample for various chemical measurements, or about 20 g from two polychlorotrifluoroethylene sample tubes may be hydrolyzed to provide the chlorine sample. The procedure for hydrolyzing the contents of the polychlorotrifluoroethylene tubes is described here.

33.1.1.1 Immerse the tubes in liquid nitrogen and cool for 10 min.

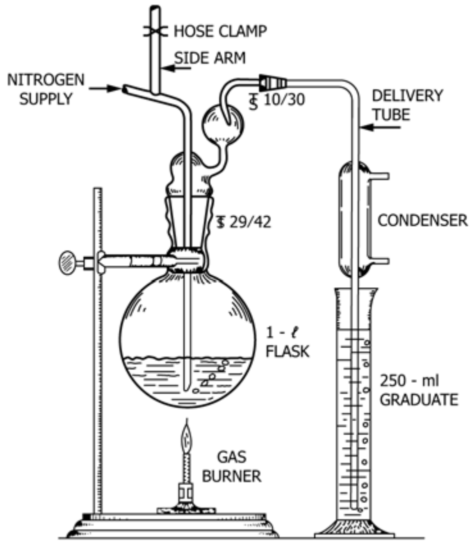


FIG. 3 Apparatus for Distillation of Chlorine

33.1.1.2 Remove the top flare plugs and collars, and place the tubes into a platinum dish or a polychlorotrifluoroethylene beaker containing 100 mL of chilled distilled water.

33.1.1.3 After hydrolysis of the UF_6, remove the polychlorotrifluoroethylene tubes and rinse with distilled water. Add the rinse solution to the UO_2F_2 solution.

33.2 *Analysis:*

33.2.1 Fill the graduated cylinder in Fig. 2 to the 150-mL level with KI-$NaC_2H_3O_2$ solution. Then connect the delivery tube so its tip is near the bottom of the solution in the receiving graduate.

33.2.2 Dispense 20 g of H_3BO_3 into the 1-L round-bottom flask.

33.2.3 Transfer the sample solution containing UO_2F_2 from approximately 20 g of UF_6 in 100 mL of solution to the flask.

33.2.4 Add 10 mL of concentrated H_2SO_4 (sp gr 1.84) to the flask, and swirl the contents for mixing.

33.2.5 Add 10 mL of the $FeSO_4$ solution, rinse the mouth of the flask, and connect the flask immediately to the apparatus as in Fig. 3.

33.2.6 Initiate nitrogen flow through the solution at a rate of 2 to 3 bubbles per second and start the water flow through the condenser.

33.2.7 Heat the contents of the flask until boiling and allow to boil for 30 s.

33.2.8 Remove the heat, add 10 mL of 1 % $KMnO_4$ solution through the sidearm, and close the sidearm by clamping the rubber tube that is attached to the end of the sidearm.

33.2.9 Reapply heat and allow the contents of the flask to simmer for 5 min.

33.2.10 Remove the heat, but continue the nitrogen purge for an additional 5 min.

33.2.11 Rinse the delivery tube into the receiving graduate and transfer the contents of the graduate to a 300-mL Erlenmeyer flask. Add 1 mL of starch solution and titrate the iodine with 0.025 *N* $Na_2S_2O_3$ solution to the starch end point. (The iodine may be measured spectrophotometrically rather than titrimetrically.)

33.2.12 Perform a blank analysis by carrying 100 mL of distilled water through procedural steps, 33.2.1 through 33.2.11, and subtracting from the sample titration.

34. Calculation

34.1 Calculate the concentration of chlorine in ppm chlorine on a uranium basis as follows:

$$Cl, ppm = (V_1 - V_2)(N)(0.03545 \times 10^4)/S$$

where:

V_1 = millilitres of thiosulfate for sample,
V_2 = millilitres of thiosulfate for blank,
N = normality of $Na_2S_2O_3$ solution, and
0.03545 = grams of chlorine per milliequivalents, and
S = grams of uranium.

35. Reliability

35.1 The precision at the 95 % confidence level is ±10 % at the 100-ppm level.

DETERMINATION OF SILICON AND PHOSPHORUS

36. Scope

36.1 Phosphorus and Silicon can be determined by ICP-MS (see Test Method C1287).

36.2 Silicon can be analyzed by Atomic Absorption without matrix separation.

36.3 Phosphorus and Silicon can be analyzed by spectrophotometry (as described below). With these procedures about 0.5 µg silicon or phosphorus per gram of uranium can be detected.

37. Summary of Test Method

37.1 The test methods are based on the development of the color known as molybdenum blue obtained by the reduction of silico- or phosphomolybdate ions.

37.2 Reduction of the silico- or phosphomolybdate ions with a combination 1-amino-2-naphthol-4-sulfonic acid, sodium sulfite, sodium pyrosulfite solution produces the same molybdenum blue complex that is measured spectrophotometrically, directly in the uranium solution at 710 nm. One gram of uranium absorbs slightly at this wavelength, but its absorbance is easily corrected by the use of an additional aliquot to which no reducing agent is added as the blank.

38. Interferences

38.1 Phosphate interference in the silicon determination is eliminated by the addition of oxalic acid to decompose any phospho-molybdate formed. Silicon in small amounts does not interfere in the phosphorus analysis since silicomolybdate does not form at the acid concentration at which the phosphomolybdic acid is formed.

38.2 Fluoride, which would be a serious interference in the silicon analysis, is complexed with boric acid. A high concentration of silicon which could interfere in the phosphorus analysis is prevented by handling hydrolyzed UF_6 samples in platinum or plastic. Other potential interferences rarely present in significant amounts are arsenic and tungsten.

39. Apparatus

39.1 *Polyethylene Bottles,* 100 and 500 mL.

39.2 *Polyethylene Beakers,* 100 mL.

39.3 *Polyethylene Pipets;* 1, 2, 5, and 10 mL.

39.4 *Spectrophotometer,* equipped with 1 and 5 cm cells as described in Practice E60.

40. Reagents

40.1 *Ammonium Hydroxide Silicon-Free*—Distill 500 mL of saturated NH_4OH through plastic tubing into 300 mL of distilled water.

40.2 *Ammonium Molybdate Solution (10 %)*—Dissolve 100 g of reagent grade $(NH_4)_6Mo_7O_{24}$ in water, and dilute the solution to 1 L with distilled water.

NOTE 4—Not all commercially available $(NH_4)_6Mo_7\,O_{24}$ is suitable. Material supplied by J. T. Baker Chemical Co. or Baker and Adamson

Products, however, has been found to be satisfactory consistently.

40.3 *Boric Acid Solution (5 %)*—Dissolve 25 g of reagent grade H_3BO_3 in water, and dilute the solution to 500 mL.

40.4 *Oxalic Acid Solution (5 %)*—Dissolve 25 g of reagent grade $H_2C_2O_4$ in water, and dilute the solution to 500 mL. (This solution is not required for determination of phosphorus.)

40.5 *Phosphorus Standard Solution (25 µg P/mL)*—Dissolve 4.6422 g of ammonium dihydrogen phosphate $[(NH_4)H_2PO_4]$ in distilled water and dilute the solution to 1 L with distilled water. Transfer 20 mL of this solution to a 1-L volumetric flask and dilute to 1 volume with distilled water to obtain a solution containing 2.0 µg P/mL.

40.6 *Reducing Mix*—Dissolve 0.1 g of 1-amino-2-naphthol-4-sulfonic acid, 1.0 g of sodium sulfite (Na_2SO_3), and 10.0 g of sodium pyrosulfite ($Na_2S_2O_5$) in water; then dilute the solution to 100 mL.

40.7 *Silicon Standard Solution (2.5 µg Si/mL)*—Dissolve 10.6 mg of precipitated silica (SiO_2) and 0.5 g of sodium hydroxide (NaOH) in a platinum dish. Transfer the solution to a 2-L plastic bottle and dilute to volume.

NOTE 5—Silicon in solution as sodium silicate is not stable when stored in polyethylene bottles. New standard solutions should be prepared monthly.

40.8 *Sulfuric Acid—Boric Acid Solution (10 % H_2SO_4—4 % H_3BO_3)*—Dissolve 20 g of reagent grade H_3BO_3 in water. Add 50 mL of concentrated H_2SO_4 (sp gr 1.84) and dilute the solution to 500 mL.

40.9 *Uranium Oxide* (U_3O_8, UO_2, or UO_3), phosphorus- and silicon-free.

NOTE 6—All standard solutions should be made and stored in plastic containers to prevent silicon contamination from glassware.

41. Procedure

41.1 *Sample Preparation:*

41.1.1 Hydrolyze a weighed portion of 6 to 10 g of UF_6 in a platinum boat in 80 mL of distilled water as described in 14.7 through 14.17.

41.1.2 Transfer the solution to a 100-mL plastic bottle and dilute to 100 mL.

41.1.3 Transfer an aliquot equivalent to 1 g of UF_6 to a 100-mL TFE-fluorocarbon beaker, and add 1 mL of 6 *N* H_2SO_4. (Phosphorus aliquots should contain 1 to 50 µg of phosphorus.)

41.1.4 Add 20 mL of 5 % H_3BO_3, and heat the solution for 20 min to complex the fluoride.

41.1.5 For phosphorus analysis only, transfer the solution to a 100-mL borosilicate beaker and evaporate to 20 mL (see 41.3).

41.2 *Determination of Silicon:*

41.2.1 *Preparation of Calibration Curve:*

41.2.1.1 Pipet standard aliquots containing 0, 2.5, 5.0, 7.5, 10.0, and 12.5 µg of silicon into plastic beakers.

41.2.1.2 Add silicon-free uranium, 0.1 g as uranyl nitrate solution, to each beaker. Prepare the uranyl nitrate solution by dissolving silicon-free uranium oxide in nitric acid in a TFE-fluorocarbon beaker.

41.2.1.3 Add from 1.0 to 1.5 mL of 18 *N* H_2SO_4 to each beaker and dilute to 25 mL. Then proceed with the analysis starting with 41.2.2.4. Plot the absorbances corrected for the blank against the known quantities of silicon taken to obtain a calibration curve. In a typical case, 10 µg of silicon gave a corrected absorbance of about 0.285 in a 5-cm cell. Up to about 150 µg can be handled using a 1-cm absorbance cell and an appropriate calibration curve.

41.2.2 *Analysis:*

41.2.2.1 A blank containing all the reagents in the amounts used in the sample aliquot must be analyzed with the samples. Normally 10 mL of 1 *N* NaOH solution gives an absorbance of 0.030 to 0.050 in this procedure. Most other reagents were found to be nearly silicon-free.

41.2.2.2 Dilute the aliquot of the sample in a 100-mL plastic beaker to 25 mL with water.

41.2.2.3 Add from 1 to 1.5 mL of 18 *N* H_2SO_4.

41.2.2.4 Place the TFE-fluorocarbon beaker containing the sample in a water bath or an oven and heat to 90 to 95°C.

41.2.2.5 Remove the beaker from the water bath or the oven, and add 5 mL of 10 % $(NH_4)_6Mo_7O_{24}$ solution immediately.

41.2.2.6 Adjust the acidity to a pH of 1.2 to 1.3 while the solution is still warm by adding silicon-free NH_4OH or HCl.

41.2.2.7 Allow the sample to stand 10 min to permit the formation of the silico-molybdate complex.

41.2.2.8 Add 10 ml of 5 % $H_2C_2O_4$ solution to the beaker and swirl. Allow the solution to stand for 2 min to decompose any phosphomolybdate.

41.2.2.9 Add 2 mL of reducing mix to the beaker and swirl.

41.2.2.10 Add sufficient 6 *N* HCl immediately to the sample to obtain a 1 *N* acid solution.

41.2.2.11 After all the precipitate is dissolved, transfer the solution to a 50 or 100-mL volumetric flask and dilute to volume with 1 *N* HCl.

41.2.2.12 Determine the absorbance of the solutions in a 5-cm cell at 710 nm.

NOTE 7—The uranyl ion shows a slight absorbance at 710 nm, and samples must be corrected for this absorbance. This is best determined by taking an additional aliquot from the sample solution and treating it as indicated in the procedure up to the point the pH is adjusted with NH_4OH. Any precipitate is dissolved with a minimum of H_2SO_4 and the solution diluted to 50 mL. The absorbance of this solution is used as an additional blank correction.

41.2.2.13 Determine the quantity of silicon in the aliquot from a previously prepared calibration curve.

41.3 *Determination of Phosphorus:*

41.3.1 *Preparation of Calibration Curve*—Using a TFE-fluorocarbon beaker, dissolve sufficient uranium oxide (phosphorus-free) containing 20 g of uranium in HNO_3. Dilute it to 200 mL in a plastic bottle. To separate 10-mL aliquots, add 0, 5, 10, 25, and 50 µg of phosphorus. Analyze by the procedure described below. Plot the absorbances corrected for the blanks against the known quantities of phosphorus to obtain a calibration curve.

41.3.2 *Analysis:*

41.3.2.1 Neutralize the sample aliquot containing 1 to 50 µg of phosphorus in a volume of 20 mL or less in a 100-mL borosilicate beaker with NH_4OH until a precipitate begins to form.

41.3.2.2 Add sufficient 6 *N* HCl to make the sample 1 *N* in acid.

41.3.2.3 Transfer the sample to a 100-mL volumetric flask and dilute to about 50 mL with 1 *N* HCl.

41.3.2.4 Add 5 mL of $(NH_4)_6Mo_7O_{24}$ solution.

41.3.2.5 Heat the sample to 90 to 95°C in a water bath.

41.3.2.6 Allow the sample to cool to room temperature in a water bath; add 2 mL of reducing mix to the solution and swirl.

41.3.2.7 Dilute the solution in the 100-mL volumetric flask to volume with 1 *N* HCl.

41.3.2.8 After 1 h, measure the absorbance of the solution in a 5-cm cell at 710 nm against a reagent blank.

NOTE 8—Since the uranyl ion absorbs slightly at this wavelength, a correction must be made for the quantity of uranium present. This can be done by measuring the absorbance of an additional untreated aliquot of the original sample made to a 50-mL volume or by applying a previously determined standard correction when the amounts of uranium in the aliquot are known (typically, each gram of uranium in a 50-mL volume gives an absorbance reading of 0.038 at 710 mm).

41.3.2.9 Determine the quantity of phosphorus present in the aliquot from a previously prepared calibration curve. Then calculate the phosphorus content of the original material on the basis desired.

42. Reliability

42.1 At the 10-µg level of either element, the precision at the 95 % confidence level is ±10 %.

DETERMINATION OF BORON AND SILICON

43. Scope

43.1 Boron and Silicon can be determined by ICP-MS (see Test Method C1287). They can also be analyzed by FTIR on gaseous UF_6 (see Test Method C1441). The test method using DC-arc spectrographic emission has been discontinued (see C761–96).

DETERMINATION OF RUTHENIUM

44. Scope

44.1 Ruthenium can be determined by ICP-MS (see Test Method C1287). The Atomic Absorption test method using MIBK extraction has been discontinued (see C761–96). The Spectrographic determination of Ruthenium has been discontinued (see C761–96).

DETERMINATION OF TITANIUM AND VANADIUM

45. Scope

45.1 Titanium and Vanadium can be determined by ICP-MS (see Test Method C1287). They can also be determined by ICP-AES as described in sections 112 to 121 and 122 to 131. The Spectrophotometric determination of these elements with BHPA-Chloroform extraction has been discontinued (see C761–96).

SPECTROGRAPHIC DETERMINATION OF METALLIC IMPURITIES

46. Scope

46.1 The Spectrographic determination of metallic impurities with BHPA or Cupferrides extraction has been discontinued (see C761–96). Most elements can be analyzed by ICP-MS (see Test Method C1287), or Atomic Absorption and ICP-AES (see further sections).

46.2 Arsenic can be analyzed with Test Method C1219 or Test Method C1287.

DETERMINATION OF TUNGSTEN

47. Scope

47.1 Tungsten can be determined by ICP-MS (see Test Method C1287) or by ICP-AES as described later in this document. The Spectrophotometric determination has been discontinued (see C761–96).

DETERMINATION OF THORIUM AND RARE EARTHS

48. Scope

48.1 Thorium and rare earths can be determined by ICP-MS (see Test Method C1287). The Spectrophotometric determination has been discontinued (see C761–04$^{\varepsilon 1}$).

DETERMINATION OF MOLYBDENUM

49. Scope

49.1 Molybdenum can be determined by ICP-MS (see Test Method C1287) or by ICP-AES as described later in this document. The Spectrophotometric determination using the thiocyanate complex has been discontinued (see C761–96).

ATOMIC ABSORPTION DETERMINATION OF METALLIC IMPURITIES

50. Scope

50.1 A test method is presented for the analysis of 14 metallic elements in uranium compounds by atomic absorption spectroscopy (**7, 8**). The test method has been shown to be applicable to the analysis of aluminum, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, sodium, and zinc.

51. Summary of Test Method

51.1 The uranium in a sample is separated by solvent extraction with tributylphosphate (TBP) from 6 to 8 *N* HNO_3 (**9**). The HNO_3 solution containing the elements to be determined is evaporated to dryness and the residue redissolved in 0.2 *N* HCl. Any or all of the elements can then be determined in this one solution by standard atomic-absorption spectrometry procedures with little or no matrix effects in the usual type of sample.

51.2 Direct atomic-absorption analysis of several elements in uranium solutions is sometimes satisfactory (**10, 11**).

51.3 The uranium is extracted with pure TBP from 6 *N* HNO_3. The aqueous layer is then washed with 20 % TBP in chloroform. A total of 99.9 % of the uranium is removed, leaving a nearly residue-free solution when the HNO_3 solution is evaporated to dryness. Since all atomic absorption analyses are then made in exactly the same medium, that is, 0.2 *N* HCl solution, standards preparation is simplified, and conditions for maximum precision and accuracy are ideal.

51.4 In relatively pure UF_6 that is usually analyzed by this test method, there is usually no problem with anionic interference. Fluoride can interfere with the extraction, but it is easily removed before the extraction by pyrohydrolysis or by fuming the sample with HNO_3. Sulfate and phosphate are possible interferences in the extraction, but they are rarely present in significant quantities.

51.5 Where more than one element is to be analyzed, the atomic absorption procedure requires substantially less labor per analysis than spectrophotometric methods. Furthermore, for some elements such as magnesium, potassium, and sodium, no satisfactory spectrophotometric methods are available. Compared to the carrier distillation spectrographic method, the atomic absorption procedure, though longer, provides better sensitivity and range, and much better precision and accuracy.

52. Apparatus

52.1 *Atomic Absorption Spectrophotometer,* as described in Proposed Recommended Practices for Atomic Absorption Spectrometry.

53. Reagents

53.1 *Carbon Tetrachloride, Reagent Grade*—Wash 1 L of CCl_4 with 100 mL of 0.1 *N* HNO_3 and then with 100 mL of deionized water.

NOTE 9—**Caution:** Carbon tetrachloride must be handled with due safety precautions. It should be handled in a hood to avoid breathing its vapor.

53.2 *Hexane, Reagent Grade*—Wash 1 L of C_6H_{14} with 100 mL of 0.1 *N* HNO_3 and then with 100 mL of deionized water.

53.3 *Hydrochloric Acid, Purified*—Distill and determine the normality as for HNO_3.[10]

53.4 *Hydrochloric Acid* (0.2 *N*)—Prepare by appropriate dilution of purified HCl with deionized water.

53.5 *Lanthanum Chloride* (*$LaCl_3$*), *Purified* (50 mg/mL)—Transfer 200 mL of saturated lanthanum nitrate [$La(NO_3)_3$], 400 mL of 2.0 *N* aluminum nitrate [$Al(NO_3)_3$], and 300 mL of purified TBP to a 1-L separatory funnel. Shake the solution vigorously for 2 min to extract lanthanum. Discard the aqueous layer, and wash the TBP phase twice with 100 mL of deionized water. Discard these washings. Transfer the TBP phase to a 1-L beaker. Add about 500 mL of calcium-free acetone or ethanol and 50 mL of water. Distill NH_4OH into this solution until precipitation of lanthanum hydroxide [$La(OH_3)_3$] is complete. Filter the precipitate and wash with deionized water. Ignite the precipitate at 800°C to form lanthanum oxide (La_2O_3). Dissolve the La_2O_3 (58.64 g) in purified HCl, and dilute to 1 L with deionized water.

53.6 *Nitric Acid (HNO_3), Purified*—Distill approximately 1700 mL of concentrated HNO_3 (sp gr 1.42) into a 2-L plastic bottle containing 400 mL of deionized water. A quartz still is preferable for the distillation, but if one is not available, borosilicate glassware is satisfactory. Determine the specific gravity of the distilled HNO_3, and calculate the normality.

53.7 *Nitric Acid* (6.0 *N)*—Prepare by appropriate dilution of purified HNO_3 with deionized water.

53.8 *Standard Stock Solution, 100 µg/mL of Each Element*—Prepare a stock solution containing all elements to be determined by dissolving 0.100 g ± 0.1 mg each of the pure metals in purified HNO_3 or HCl. Convert the nitrate solutions to chloride when feasible by digestion with HCl. Add lead as the nitrate. Dilute the stock solution to 1 L with 0.2 *N* HCl. After a preliminary analysis has been made on an unknown sample to determine the approximate concentration of impurities, dilute aliquots of the stock solution to give concentrations that bracket the sample. Alternatively use certified reference standards.

53.9 *n-Tributylphosphate (TBP), Purified*—Wash 500 mL of purified TBP with at least four 500-mL portions of deionized water to remove sodium and orthophosphate.

NOTE 10—For determining low concentrations of sodium (for example, <5 µg/g U), special purification steps such as vacuum distillation in a quartz system may be required.

53.10 *Urano-Uranic Oxide (U_3O_8)*—Hydrolyze redistilled UF_6 in deionized water to form uranyl fluoride (UO_2F_2). Evaporate the solution to dryness in a plantinum dish and pyrohydrolyze the residue at 900°C for 6 to 8 h with occasional mixing to form U_3O_8.

54. Procedure

54.1 Hydrolyze enough UF_6 to contain the desired amount of uranium (5 to 20 g) with deionized water.

54.2 Evaporate the solution to dryness under heat lamps, and convert the UO_2F_2 to U_3O_8 by pyrohydrolysis at 900°C for 45 min. (U_3O_8 produced in the gravimetric analysis of uranium in UF_6 is suitable.)

NOTE 11—If elements that form volatile fluorides are to be analyzed by atomic absorption, the UO_2F_2 solution should be digested with HNO_3 several times to remove fluoride and obtain a suitable $UO_2(NO_3)_2$ solution for the extraction.

54.3 Weigh enough U_3O_8 to give the desired weight of uranium (to the nearest milligram) into a platinum dish or a TFE-fluorocarbon beaker.

54.4 Dissolve the sample in purified concentrated HNO_3 (sp gr 1.42, 1 mL acid per gram of oxide), and evaporate the solution to near dryness.

NOTE 12—When uranium fluorides containing chromium are pyrohydrolyzed, a nitric acid-insoluble compound, probably $CrUO_4$, is formed. Consequently, samples to be analyzed for chromium must be fumed with perchloric acid ($HClO_4$) *after* dissolution with HNO_3 to dissolve this uranium-chromium compound. Add a few drops of 30 %

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[10] Alternatively, high purity/distilled acids may be purchased from a vendor.

hydrogen peroxide (H_2O_2) to reduce chromium (VI) to chromium (III) before extraction of the uranium.

54.5 Dissolve the residue in 6 *N* HNO_3, and dilute the solution to 100 mL with 6 *N* HNO_3.

NOTE 13—Blanks containing all reagents *must* be run through the entire procedure.

54.6 Transfer the sample solution containing up to 10 g of uranium to a 250-mL separation funnel, using 6 *N* HNO_3 to rinse the beakers. Use a 500-mL separation funnel for samples containing 10 to 20 g of uranium. (Plastic separation funnels are preferred.)

54.7 Add 50 mL of purified TBP to the separation funnel for each 4 g of uranium.

54.8 Shake the separation funnel vigorously for 2 min to extract the uranium.

54.9 Allow the phases to separate completely (this requires about 15 min).

NOTE 14—When the extracted sample contains 15 to 20 g uranium, the density of the TBP phase is greater than the density of the aqueous phase. Add 50 mL hexane *after the extraction* to reduce the density of the TBP phase. (If the hexane were added before the extraction, the resulting TBP-hexane mixture would have a lower extraction efficiency.)

54.10 Transfer the aqueous phase to a second separation funnel. Wash the TBP phase with two 30-mL portions of 6 *N* HNO_3, and add the washings to the second separation funnel.

54.11 Wash the aqueous phase with 50 mL of 20 % TBP in CCl_4. After separation, drain off the organic phase.

54.12 Wash the aqueous phase twice with 25-mL portions of CCl_4.

54.13 Transfer the aqueous phase to a TFE-fluorocarbon beaker or platinum dish, and evaporate the solution to dryness.

54.14 Dissolve the residue in 0.2 *N* HCl and dilute to a desired volume according to the following tabulation:

Impurity, µg/g U	Wt of Uranium, g	Volume, mL
0.1–0.5	20	10
0.5–1	20	25
1–5	10	25
5–50	10	50–100
>50	5	100

54.15 Determine the desired elements by standard atomic absorption techniques, comparing the sample measurements to those of known standards in the same concentration ranges.

NOTE 15—Samples to be analyzed for calcium or magnesium require the addition of lanthanum or strontium to eliminate suppression. Pipet an aliquot of the sample into a volumetric flask, and add sufficient lanthanum or strontium chloride to give 10 mg of lanthanum or strontium per mL. Dilute the solution to volume with 0.2 *N* HCl and analyze by comparison to known standards also containing lanthanum or strontium.

54.16 Table 4 gives the operating parameters for the atomic-absorption analysis of the elements, using an atomic-absorption spectrometer equipped with a Boling burner for all elements except aluminum, which must be analyzed with the nitrous oxide burner. Adjust the burner settings for a maximum absorption with copper and leave at those settings for the rest of the analyses.

TABLE 4 Operating Parameters for the Atomic-Absorption Analysis of the Elements

Element	Concentration Range, μ g/mL	Wavelength, nm	Recommended Flows	
			Fuel	Air
Al	10–200	309.27	>15[A]	8 (N_2O)
Ca	0–20	422.67	≈9[A]	7.5
Cd	0.5–5	228.80	9	9
Co	4–20	240.73	9	9
Cr	2–20	357.87	≈9[A]	7.5
Cu	2–20	324.75	9	9
Fe	2–20	248.33	9	9
K	1–10	766.48	9	9
Mg	0–2	285.21	≈9	7.5
Mn	2–20	279.48	9	9
Na	0.3–3	589.00	9	9
Ni	2–25	232.00	9	9
Pb	0–40	217.00	9	9
Zn	0–5	213.86	9	9

[A] Fuel adjusted to maximum percentage of absorption.

55. Reliability

55.1 At the 1 to 10 µg/g U level, all the elements except aluminum can be determined with a precision of ±10 % at the 95 % confidence interval. The precision for the aluminum analysis is ±30 % at the 95 % confidence interval.

IMPURITY DETERMINATION BY SPARK SOURCE MASS SPECTROGRAPHY

56. Scope

56.1 The spark-source mass-spectrographic technique has been discontinued (see C761–96).

DETERMINATION OF BORON-EQUIVALENT NEUTRON CROSS SECTION

57. Scope

57.1 The determination of boron equivalent neutron cross section can be found in Practice C1233.

DETERMINATION OF URANIUM-233 ABUNDANCE BY THERMAL IONIZATION MASS SPECTROMETRY

58. Scope

58.1 The determination of uranium-233 has been discontinued (see C761–96). Uranium-233 analysis could be performed referring to Test Method C1413. However, the method should be adapted for the determination of this isotope.

DETERMINATION OF URANIUM-232 BY ALPHA SPECTROMETRY

59. Scope

59.1 Uranium-232 can be determined using Guide C1636.

59.2 This test method is applicable to the determination of uranium-232 in uranyl fluoride solutions, in concentrations as low as 0.05 ppb ^{232}U/U.

60. Summary of Test Method

60.1 Uranyl fluoride solutions are evaporated to dryness, and the uranium is converted to the oxide. A weighed portion of the oxide is dissolved in HNO_3 and electroplated on a stainless steel disk. The alpha activities from ^{232}U with energies of 5.28 and 5.32 MeV and ^{228}Th with energies of 5.34 and 5.42 MeV are measured with a pulse height analyzer. The two ^{232}U energy peaks are summed and corrected for the unresolved ^{228}Th 5.34 MeV. The counts are converted to disintegration rate and divided by the specific alpha activity of ^{232}U to determine the weight of ^{232}U on the disk.

61. Apparatus

61.1 *Multiple-cell Electroplating Apparatus* (**12**) with four cells operating independently of each other and the current within each cell automatically controlled to 3 A at 32 V dc. The speed of the stirrers shown in Fig. 4 is 500 rpm.

61.2 *Silicon Surface Barrier Detector,* or equivalent.

61.3 *Multichannel Alpha Spectrometer* with surface-barrier detector. For a detailed description of Alpha Spectrometry, see Test Method D3084.

62. Reagents

62.1 *Ammonium Oxalate Solution,* (0.4 *M*)—Dissolve 56.8 of $(NH_4)_2C_2O_4·H_2O$ in warm distilled water and dilute to 1 L.

62.2 *Gas Mixture*—Ionizing gas for Frisch grid ionization chamber; 90 % argon, 10 % methane.

62.3 *Plutonium Standard*—^{238}Pu and ^{239}Pu, with an activity approximately 10 000 cpm, deposited on a 52-mm diameter stainless steel disk.

62.4 *Neptunium Standard*—^{237}Np on a 25 mm diameter stainless steel disk for an alpha activity of 833 to 1667 Bq. The activity on the disk should be calibrated such that it is traceable to national or international standards; for example, in the U.S., standards maintained by the National Institute of Standards and Technology (NIST).

63. Procedure

63.1 *Sample Preparation:*

63.1.1 Evaporate the uranyl fluoride (UO_2F_2) solution, obtained by hydrolysis of a UF_6 subsample, to dryness; ignite to U_3O_8 in a platinum dish; and weigh the oxide.

63.1.2 Dissolve a sample containing 25 mg of uranium in 1 mL of 8 *N* HNO_3 and dilute to 500 mL.

63.1.3 Place a nickel disk (Grade A, cold-rolled, smooth finish, 52–mm diameter) in the center depression of the electroplating cell base plate, and a rubber gasket on a polychlorotrifluoroethylene or glass cell stack. (High-luster 300 series stainless steel can be substituted for nickel.)

63.1.4 Place the cell stack and gasket, as a unit, on a nickel or stainless steel disk which serves as the bottom and cathode of the electroplating cell.

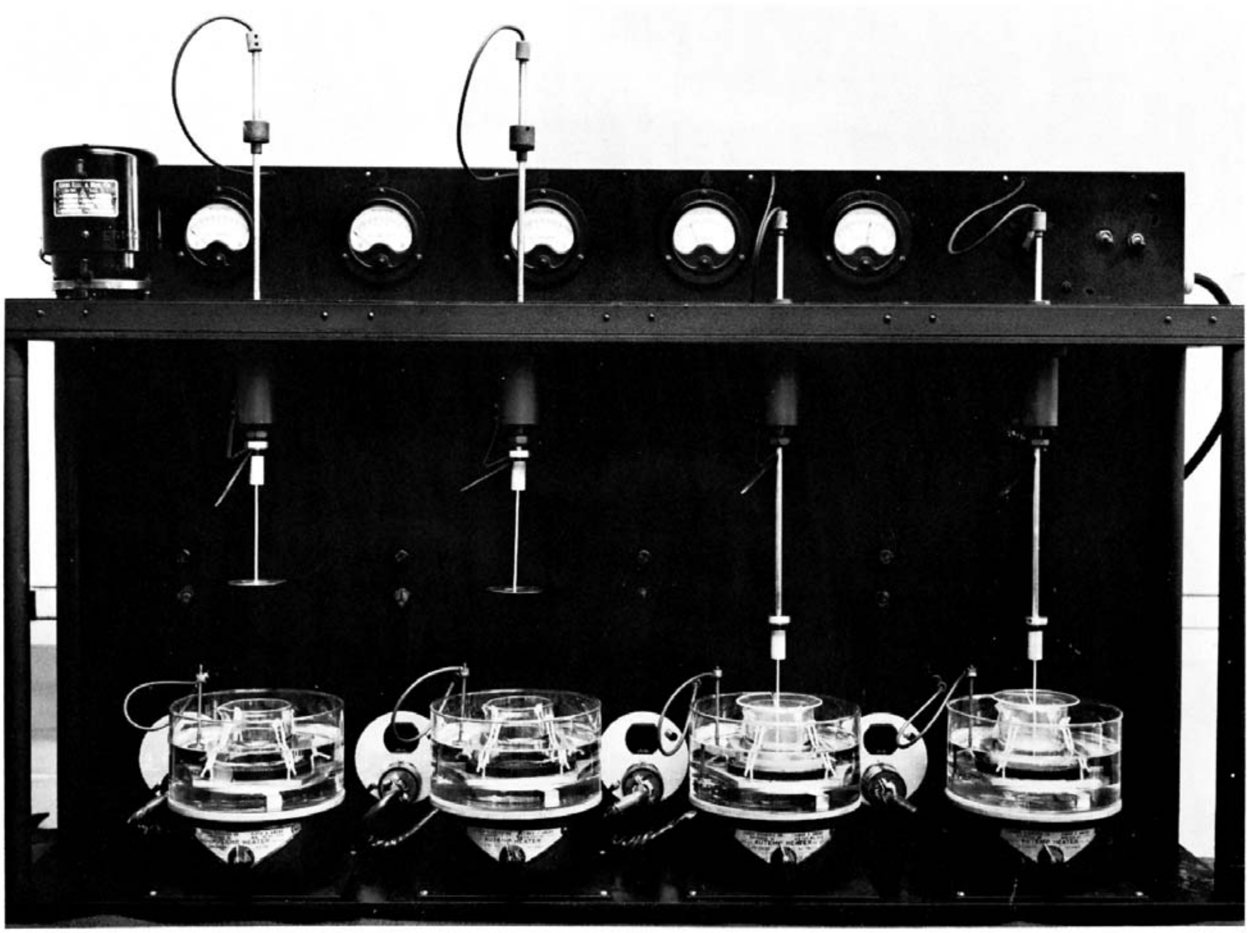

FIG. 4 Multiple-Cell Electroplating Apparatus

63.1.5 Fasten the cell stack to the base plate, making a leak-proof seal between the disk and the cell stack.

63.1.6 Add 10 mL of 0.4 *M* $(NH_4)_2C_2O_4$ solution to the cell, then add a volume of sample containing 0.5 mg of uranium.

63.1.7 Adjust the volume of the solution to 25 mL with distilled water.

63.1.8 Place the cell in a water bath, between 75 and 85°C, on the electroplating apparatus.

63.1.9 Turn on the electroplating apparatus, and lower the platinum anode into the solution until the anode is about 10 mm above the disk.

63.1.10 Add distilled water to replace the water evaporated during electroplating.

63.1.11 After 45 min, remove the cell from the electroplating apparatus, and quickly pour out the electrolyte.

63.1.12 Rinse the cell with approximately 15 mL of ethyl alcohol to dry the film.

63.1.13 Disassemble the cell stack, and heat the uranium on the disk in a furnace at 425°C for 10 min.

63.2 *Counting:*

63.2.1 Place the ^{238}Pu and ^{239}Pu standard in the surface barrier detector chamber under the active area of the detector.

63.2.2 Connect the surface-barrier detector through the preamplifier and amplifier to the analyzer.

63.2.3 Turn on the vacuum pump connected to the detector chamber, and pump the chamber to a pressure of approximately 2 Pa.

63.2.4 Adjust the detector bias voltage to the voltage specified for the detector.

63.2.5 Adjust the amplifier to cover a spectrum area approximately 3.7 to 7.0 MeV, and measure the alpha emissions 10 min to determine the resolution at 5.14 and 5.48 MeV. The resolution must not exceed 0.050 MeV.

63.2.6 Turn the bias voltage to zero.

63.2.7 Close the vacuum line, vent the detector chamber, and remove the plutonium standard.

63.2.8 Place the neptunium standard under the detector; adjust the vacuum and bias voltage as in 63.2.3 and 63.2.4, and alpha count 20 min to determine the counter efficiency factor. The activity of the standard has been determined on a parallel-plate alpha counter of known counter efficiency.

63.2.9 Remove the neptunium standard as in 63.2.7.

63.2.10 Place the uranium sample under the detector; adjust the vacuum and bias voltage as in 63.2.3 and 63.2.4, and count the sample 40 min. Lower the counting time if the sample contains a significant amount of ^{232}U.

63.2.11 Obtain the sum of all counts in the 5.3 MeV peak (which includes the ^{232}U at 5.32 and 5.28 MeV plus the unresolved ^{228}Th at 5.34 MeV). Also, determine the sum of the ^{228}Th counts in the 5.42–MeV peak.

64. Calculation

64.1 Convert total counts to net ^{232}U counts per minute as follows:

$$^{232}C = (C_{5,3} - 0.394C_{5,42})/t \tag{3}$$

where:

^{232}C = net ^{232}U counts per minute,

$C_{5,3}$ = total counts in 5.32 and 5.27 MeV of ^{232}U peak and 5.34–MeV peak of ^{228}Th corrected for background,

$C_{5,42}$ = ^{228}Th counts in 5.42–MeV peak corrected for background,

t = time in minutes for $C_{5,3}$ and $C_{5,42}$, and

0.394 = known ratio of ^{228}Th counts at 5.34 MeV to counts at 5.42 MeV.

64.2 Determine the ^{232}U disintegration rate of the sample as follows:

$$D = {}^{232}C/E \tag{4}$$

where:

D = sample disintegrations per minute, and

E = counter efficiency obtained by dividing the net counting rate obtained on a neptunium standard by the known disintegration rate for the standard.

64.3 Calculate the amount of ^{232}U in nanograms per gram of ^{235}U as follows:

$$^{232}\text{U}/\,^{235}\text{U, ng/g} = D/(4.65 \times 10^4 \times W \times F) \tag{5}$$

where:

D = sample disintegrations per minute,

4.65×10^4 = alpha activity of ^{232}U in disintegrations per minute per nanogram,

W = weight of sample aliquot counted, g U, and

F = weight fraction ^{235}U in sample.

65. Reliability

65.1 A95 % confidence limit of ±16 % for a single determination has been obtained by analyzing eight separate aliquots from a uranium solution containing 100 µg/g ^{232}U/^{235}U.

DETERMINATION OF FISSION PRODUCT ACTIVITY

66. Scope

66.1 The gamma activity of fission products can be determined using Test Method C1295. The beta activity of fission products can be determined according to Test Method C799 by beta counting after separation of uranium with TBP.

DETERMINATION OF PLUTONIUM BY ION EXCHANGE AND ALPHA COUNTING

67. Scope

67.1 This test method **(13)** provides for the efficient carrier-free separation of plutonium activity from uranium. The separated plutonium activity can then be determined by alpha counting. Plutonium alpha activities of 5 Bq/g of uranium can be detected, and at the 50 Bq/g of uranium level the method has a precision of about 15 to 20 %.

68. Summary of Test Method

68.1 Plutonium is commonly separated from uranium and most other elements by precipitation with fluoride using lanthanum as a carrier **(14).** For uranium of high-specific alpha activity (for example, more than 1 % ^{234}U or ^{233}U in isotopic composition), this method does not give satisfactory separation from uranium alpha activity. When this precipitation procedure is combined with the anion exchange procedure described by Wish and Rowell (**15**), a complete separation from uranium activity is achieved, and the plutonium is provided in a carrier-free residue that gives excellent counting characteristics.

68.2 Since the method combines two separation processes, based on entirely different principles, elements interfering in one are not likely to interfere in the other. Elements that can be carried down with plutonium in the flocculent lanthanum fluoride precipitate include: barium, neptunium, and thorium. Uranium is not precipitated but is carried along mechanically.

68.3 Of these elements, only neptunium and uranium will absorb along with the plutonium from 12 *M* HCl on a strongly basic anion-exchange resin. Barium and thorium will not absorb. Of the absorbed elements, only the plutonium is reduced and removed with the eluting agent; that is, ammonium iodide and hydroxylamine hydrochloride in 12 *M* HCl.

68.4 Evaporation of this eluate with HNO_3 leaves behind a very small, adhesive residue that is ideally suited to alpha counting.

69. Apparatus

69.1 *Ion-Exchange Column*—The bottom half of a 10-mL pipet serves satisfactorily for this purpose.

69.2 *Proportional Counter.*

69.3 *Motor-Driven Stirrer,* made from a platinum wire sealed in a glass rod.

70. Reagents

70.1 *Dowex-1, X-8,* 500 to 100 mesh.

70.2 *Hydroxylamine Hydrochloride* (4 *M*)—Prepare a 4 *M* solution of hydroxylamine hydrochloride ($NH_2OH·HCl$).

70.3 *Lanthanum Nitrate Solution* (0.05 *M*)—Prepare a 0.05 *M* solution of lanthanum nitrate ($La(NO_3)_3$).

70.4 *Nitric Acid-Hydrofluoric Acid Wash Solution* (1 *N* HNO_3 and 5 *N* HF).

70.5 *Plutonium Nitrate [$Pu(NO_3)_6$] Standard Solution,* pure, of known alpha activity of about 100 Bq/mL.

70.6 *Potassium Hydroxide* (KOH) 50 %, carbonate-free.

70.7 *Reducing Solution*—Concentrated HCl (sp gr 1.19), saturated with $NH_2OH·HCl$; the resulting solution is made 0.1 *M* with respect to ammonium iodide (NH_4I).

71. Procedure

71.1 Fume an aliquot containing up to 0.1 g of uranium with concentrated H_2SO_4 (sp gr 1.84) to remove the fluoride ion and nitrate ion.

71.2 Transfer the aliquot to a 15-mL centrifuge tube.

71.3 Add 2 mL of $NH_2OH·HCl$ solution, and heat the solution for 20 min at 80°C.

71.4 Add 1 mL of the $La(NO_3)_3$ solution, and mix the plutonium solution thoroughly.

71.5 Add 1 mL of 1 + 1 HF and stir.

71.6 Centrifuge the mixture for 2 min, and decant the supernatant liquid. Wash the precipitate with 1 mL of HNO_3-HF solution with the aid of the motor-driven stirrer. Then centrifuge the solution, decant the supernate, and repeat the washing process.

71.7 Add 1 mL of KOH solution, and wash the precipitate. Centrifuge the solution for 2 min, and decant the supernatant part. Wash the precipitate and centrifuge again with the caustic solution, and then wash once with water to remove excess KOH.

71.8 Dissolve the precipitate with 2 to 3 mL of concentrated HCl (sp gr 1.19) containing a trace of HNO_3 [one drop of concentrated HNO_3 (sp gr 1.42) per 15 mL of concentrated HCl (sp gr 1.19)].

71.9 Prepare an ion-exchange column with Dowex-1 by pouring enough resin slurry into the column to give a resin bed about 15 cm high. Pass 10 mL of concentrated HCl (sp gr 1.19) through the column.

71.10 Pass the solution of the dissolved precipitate through the ion-exchange column at 4 drops/min (a slight vacuum will be required to maintain this rate of flow). Wash the resin with 5 mL of concentrated HCl (sp gr 1.19).

71.11 Elute the absorbed plutonium with 10 mL of reducing solution.

71.12 Add 2 to 3 drops of concentrated HNO_3 (sp gr 1.42) to the eluate, and evaporate it to dryness. Dissolve the residue with 2 to 3 drops of concentrated HNO_3 (sp gr 1.42), and rinse the sides of the beaker with 1 mL of water.

71.13 Transfer the solution to a counting planchet and evaporate to near dryness. Rinse the beaker twice with 1 mL of water; transfer the washings to the planchet and evaporate to dryness.

71.14 Alpha count the residue on a proportional counter.

71.15 Convert the alpha counts to disintegrations using a geometry and recovery factor determined by analyzing known amounts of plutonium activity by the above procedure. Recoveries of 90 to 100 % of 600 dis/min of plutonium activity should be achieved.

DETERMINATION OF PLUTONIUM BY EXTRACTION AND ALPHA COUNTING

72. Scope

72.1 This thenoyltrifluoroacetone (TTA) test method covers the determination of total plutonium in UF_6. Plutonium can be quantitatively and selectively extracted from an aqueous solution into a TTA-xylene solution. Plutonium can also be determined using Guide C1561.

73. Summary of Test Method

73.1 Plutonium-bearing UF_6 is hydrolyzed using a nitric acid-aluminum nitrate solution. The plutonium is then reduced with hydroxylamine hydrochloride to Pu^{+3}, oxidized to Pu^{+4} with sodium nitrite, and extracted into TTA. Removal from

TTA is with nitric acid. The plutonium-bearing aqueous phase is then evaporated to dryness on appropriate surfaces for counting gross alpha. Counting rates are compared to those of known standards to determine total plutonium. A tracer may also be used to check the recovery rate after the extraction. ^{236}Pu has been found suitable.

74. Interferences

74.1 There is no appreciable interference due to uranium and thorium; however, neptunium is not quantitatively separated from the plutonium in the extraction procedure. If prepared sample disks have appreciable alpha counts, an alpha energy analysis should be performed and a neptunium correction applied when necessary. For samples counting near the detectability level, the alpha energy scan is useless since sensitivity is inadequate to distinguish between neptunium and plutonium.

75. Apparatus

75.1 *Alpha Counter* with a background counting rate of 5 cpm or less is recommended. Either a proportional counter or parallel-plate alpha counter is suitable.

75.2 *Alpha Energy Analyzer* is necessary for checking the selectivity and recovery rate of the extraction process.

75.3 *Equipment for Agitating Solutions* is desirable. A variable-speed laboratory shaker or a bank of extraction cells and stirrers will suffice.

76. Reagents

76.1 *Thenoyltrifluoroacetone (TTA) Solution (0.5 M)*—Dissolve 111 g of TTA in 1 L of xylene.

76.2 *Hydroxylamine Hydrochloride Solution* (1*M*)—Dissolve 69.5 g of $NH_2OH·HCl$ in 1 L of water.

76.3 *Sodium Nitrite Solution* (1*M*)—Dissolve 69 g of $NaNO_2$ in 1 L of water (prepare daily).

76.4 *Aluminum Nitrate* (2 *M*)—Dissolve 187.5 g of $Al(NO_3)_3·9H_2O$ in 250 mL of 2 *M* HNO_3.

76.5 *Nitric Acid* (6 *M*)*-Aluminum Nitrate* (0.1 *M*) *Solution*—Add 375.5 mL of HNO_3 and 37.51 g of $Al(NO_3)_3$ to 1 L of water.

77. Procedure

77.1 *Hydrolysis:*

77.1.1 Hydrolyze a sample aliquot containing 5 g of uranium as UF_6 using 250 mL of 6 *M* HNO_3-0.1 *M* $Al(NO_3)_3$ solution.

77.1.2 Hydrolyze a standard of plutonium-free UF_6 with the above solution, using 50 mL of the hydrolyzing solution per gram of uranium.

77.2 *Extraction:*

77.2.1 Transfer duplicate 50 mL aliquots from the prepared sample solution to 150 mL beakers. (For each group of samples, prepare a blank and a spike solution from the plutonium-free uranium standard. The blank is a 50 mL aliquot of the uranium standard in a 150 mL beaker. The spike is a similar aliquot spiked with 40 Bq plutonium.)

77.2.2 Evaporate sample, blank, and spike solutions to dryness slowly on a hot plate and treat identically throughout the remainder of the procedure.

77.2.3 Flame the solid residue to eliminate fluorides.

77.2.4 Use approximately 10 mL of 2 *N* HNO_3 to put the solid residue back in solution.

77.2.5 Add 4 mL of 2 *M* $Al(NO_3)_3$ – 2 *M* HNO_3.

77.2.6 Add 2 mL of 1 *M* $NH_2OH·HCl$ solution.

77.2.7 Stir the solution and allow to stand in a water bath at 80°C for 5 min.

77.2.8 Remove the sample from the water bath, and add 8 mL of 1 *M* $NaNO_2$ solution cautiously. Stir the solution, allow to stand for 5 min, and then transfer to an extraction cell.

77.2.9 Add 20 mL of 0.5 *M* TTA solution in xylene, and stir the solution for 15 min.

77.2.10 Discard the aqueous phase.

77.2.11 Wash the organic phase four times with approximately 15 mL of 2 *M* HNO_3. Perform the washings by adding wash solution, stirring a few seconds, and discarding the aqueous phase. As an alternative to the re-extraction of Pu in the aqueous phase, a direct deposition of the organic phase may be performed.

77.2.12 Add 4 mL of 8 *M* HNO_3 to the organic phase. (8 *M* HNO_3 may be replaced with 0.6 *M* HF for extracting plutonium out of the organic phase.)

77.2.13 Stir the solution for 15 min.

77.2.14 Withdraw the aqueous phase containing plutonium.

77.2.15 Pipet a 1-mL aliquot of plutonium-bearing solution onto a stainless steel alpha-counting disk, and evaporate to dryness under a heat lamp. (Stainless steel disks may be replaced by stainless steel dishes.)

77.2.16 Heat the disk over open flame to red heat and cool.

77.3 *Counting:*

77.3.1 Count the sample, blank, and spike disks for gross alpha.

77.3.2 In case of doubt concerning selectivity of extraction, perform an alpha energy scan to assure that the sample count is due to plutonium.

78. Calculations

78.1 Since each sample aliquot contains 1 g of uranium, the following expressions hold:

$$\text{Pu alpha cpm/gU} = A_0/GF \times (S - B)/(A_s - B) \quad (6)$$

where:

A_O = alpha disintegrations per minute in spike aliquot,
GF = geometry factor,
S = alpha cpm from sample disk,
B = alpha cpm from blank disk, and
A_s = alpha cpm from spike aliquot disk.

78.2 Plutonium alpha activity in disintegrations per minute per gram of uranium may be obtained by multiplying the result in Eq 6 by a geometry factor that is found by counting a plutonium standard of known disintegration rate. With most plates, this factor is 2.

78.3 Calculate parts per billion plutonium as follows:

$$\text{ppb Pu} = ((\text{Pu alpha cpm})/\text{gU})/((136)\ /\text{GF}) \quad (7)$$

where:

ppb Pu = parts per billion plutonium on a uranium basis, and

136 = specific activity for one nanogram of ^{239}Pu (cpm/ng).

79. Reliability

79.1 The procedure as described has a 95 % symmetrical confidence level of ±10 % at alpha rates greater than about 136 dpm/g uranium.

79.2 By using larger sample aliquots and plating more than 1 mL of extracted solution, concentrations as low as 8 dpm/g of uranium may be measured to a 95 % symmetrical confidence interval of ±20 % of the value.

DETERMINATION OF NEPTUNIUM BY EXTRACTION AND ALPHA COUNTING

80. Scope

80.1 The thenoyltrifluoroacetone (TTA) test method covers to the determination of ^{237}Np in UF_6. Neptunium can be selectively extracted from an aqueous solution into a TTA-xylene solution, and a ^{239}Np tracer technique can be used to measure extraction losses; thereby, eliminating the need for a more laborious quantitative extraction. Neptunium can also be determined using Guide C1561.

81. Summary of Test Method

81.1 Neptunium-bearing UF_6 is hydrolyzed using a nitric acid-aluminum nitrate solution. The resulting solution is spiked with a ^{239}Np tracer, brought to dryness, and flamed to rid the residue of fluorides. Residue is dissolved with hydrochloric acid, the neptunium reduced to Np^{+4}, and extracted into TTA. Neptunium is recovered from TTA in nitric acid as the Np^{+5} ion. The neptunium-bearing solution is evaporated to dryness on appropriate counting disks, and the necessary counting is performed.

82. Interferences

82.1 There is no radiochemical interference of consequence. Uranium, thorium, and plutonium are essentially removed in the extraction procedure. An alpha energy scan is optional to preclude interference.

83. Apparatus

83.1 *Alpha Counter* with a background counting rate less than 5 cpm is recommended. Either a proportional counter or a parallel-plate alpha counter is suitable.

83.2 *Gamma Scintillation Spectrometer,* required for the ^{239}Np tracer. A single-channel analyzer is adequate, with a multichannel instrument being optional.

83.3 *Alpha Energy Analzyer,* optional for checking the selectivity of the extraction process.

83.4 *Equipment for Agitating Solutions,* desirable. Extraction cells or separation funnels will suffice.

84. Reagents

84.1 *Hydrochloric Acid* (1 *M*)—Prepare a 1 *M* solution of hydrochloric acid (HCl).

84.2 *Nitric Acid* (HNO_3) (6*M*)—*Aluminum Nitrate* [$Al(NO_3)_3$] (0.1 *M*) *Solution.*

84.3 *Reducing Solutions*—150 mL of 5 *M* hydroxylamine hydrochloride ($NH_2OH·HCl$) + 250 mL of 2 *M* hydrochloric acid (HCl) + 100 mL of 1.5 *M* ferrous chloride ($FeCl_2$). The solution is unstable, therefore, store it in a dark bottle and prepare every two weeks.

84.4 *Thenoyltrifluoroacetone* (*TTA*) (0.5 *M*)—Dissolve 111 g of TTA in 1 L of xylene solution.

85. Procedure

85.1 *Preparation of ^{239}Np Tracer (see note below):*

NOTE 16—^{239}Np can also be obtained from a purchased ^{243}Am solution. Np and Am can be separated on a ion exchange resin.

85.1.1 Encapsulate aliquots of 100 mg normal or depleted U_3O_8 in high-silica ampules and expose for 10 min to a nominal neutron flux of 2×10^{14} n/cm^2·s.

85.1.2 Break the ampules and put the contents in solution with 6 *M* HNO_3.

85.1.3 The extraction procedure for ^{239}Np tracer is the same as that for extracting ^{237}Np from sample solutions (see 85.3).

85.1.4 With a ^{239}Np half-life of 2.3 days, the usable life of a batch of tracer is about 2 weeks. (The tracer technique gives greater accuracy and precision with a minimum of analytical effort; however, the analysis can be performed without ^{239}Np. If tracer is not used, ^{237}Np standards should be run through the extraction procedure to determine a loss correction and the procedural steps performed methodically to assure uniform losses.)

85.2 *Preparation of Sample:*

85.2.1 Hydrolyze a sample aliquot containing 5 g of uranium as UF_6 using 250 mL of 6 *M* HNO_3-0.1 *M* $Al(NO_3)_3$ solution.

85.2.2 Transfer duplicate 50-mL aliquots from the hydrolyzed solution to 150-mL beakers.

85.2.3 Add 2 mL of tracer solution to each aliquot (adjust the concentration of ^{239}Np tracer solution by appropriate dilution to give about 1000 cpm/mL when the scintillation counter is accepting photons from the 0.28-MeV gamma peak).

85.3 *TTA Extraction:*

85.3.1 Bring the spiked sample aliquots to dryness slowly on a hot plate to prevent spattering.

85.3.2 Heat each residue over an open flame until it becomes burnt orange color to remove fluorides and nitrates.

85.3.3 Cool the residue and put in solution with approximately 30 mL of 1 *M* HCl.

85.3.4 Add approximately 15 mL of reducing solution, and allow the solution to digest for 5 to 10 min.

85.3.5 Transfer the sample to an extraction cell, add 15 mL of TTA-xylene, and stir the resulting mixture for 20 min. (Replace xylene by benzene if desired.)

85.3.6 Discard the aqueous phase.

85.3.7 Wash the organic phase three times with 1 *M* HCl. Wash by adding HCl, stir ½ min, and discard the aqueous phase.

85.3.8 Add 5 to 10 mL of 8 *M* HNO_3 to the organic phase, and stir the resulting mixture for 20 min.

85.3.9 Withdraw the aqueous phase, containing neptunium, and bring to dryness on a hot plate.

85.3.10 Repeat 85.3.3 through 85.3.7.

85.3.11 Wash the organic phase twice as in 85.3.7 except with 0.05 M HNO_3.

85.3.12 Add 4 mL of 8 M HNO_3 to the organic phase, and stir the resulting mixture for 20 min.

85.3.13 Withdraw the aqueous phase, containing neptunium.

85.4 *Sample and Tracer Disk Preparation:*

85.4.1 *Sample Disk:*

85.4.1.1 Pipet 2 mL of the sample solution onto a stainless steel disk and allow to dry under a heat lamp.

85.4.1.2 Heat the disk to red heat over an open flame and cool.

85.4.2 *Tracer Disk:*

85.4.2.1 Pipet 1 mL of tracer solution onto a stainless steel disk and allow to dry under a heat lamp.

85.4.2.2 Heat the disk to red heat over an open flame and cool.

85.5 *Counting:*

85.5.1 Count the sample disks to determine net alpha counts per minute.

85.5.2 Count the sample and tracer disks to determine net gamma activity (counts/min) due to the 0.28 MeV ^{239}Np peak.

85.5.3 An alpha energy scan is optional to certify that all alpha activity is due to ^{237}Np.

86. Calculations

86.1 Each sample aliquot contains 1 g of uranium. If A equals the net ^{237}Np alpha count per minute on the sample disk, B equals the net gamma count of the ^{239}Np spike, and C equals the net ^{239}Np gamma count extracted; the following equation gives ^{237}Np alpha concentration in sample.

$$^{237}\text{Np alpha cpm/gU} = AB/C \tag{8}$$

86.2 Neptunium alpha activity in disintegrations per minute per gram of uranium may be obtained by multiplying the result in Eq 8 by a geometry factor that is found by counting a neptunium standard of known disintegration rate. With most standard plates or disks, this factor is 2. Calculate as follows:

$$\text{ppm Np} = ((\text{Np alpha cpm})/\text{gU})/((1562)/(GF)) \tag{9}$$

where:

ppm Np = parts per million neptunium on a uranium basis,
1562 = specific activity for 1 µg of ^{237}Np (cpm/µg), and
GF = geometry factor.

87. Reliability

87.1 The procedure has a 95 % symmetrical confidence interval of ± 10 % at alpha rates greater than about 2.6 Bq/g of uranium.

87.2 At 16 dpm/g uranium, the confidence interval is ± 30 %.

87.3 The lower limit of detection is about 0.067 Bg/g of uranium.

ATOMIC ABSORPTION DETERMINATION OF CHROMIUM SOLUBLE IN URANIUM HEXAFLUORIDE

88. Scope

88.1 A test method is presented for the determination of chromium, soluble in UF_6, in the concentrations of 0.2 to 100 µg/g (uranium basis).

89. Summary of Test Method

89.1 The UF_6 is filtered through a porous filter (see Practice C1689), and the filtered sample is hydrolyzed in deionized water. The chromium in the hydrolyzed UF_6 solution is separated from the uranium, with or without prior concentration, with an *n*-tributyl phosphate (TBP)-xylene mixture, leaving the chromium in the aqueous phase. The chromium is then determined by atomic absorption spectroscopy after dilution to a standard volume. Combining atomic absorption spectroscopy with solvent extraction of the chromium has two advantages: (*1*) uranium matrix effects are eliminated from the atomic absorption spectrometry and (*2*) radioactivity contamination problems arising from aspirating uranium solutions are eliminated.

89.2 Two extraction techniques are presented. In Method A, a sample solution containing 1 g of uranium is used and does not include a concentration step prior to extraction. Therefore, the detection limit is not as low as Method B that includes a 5 g uranium sample and a concentration step prior to the extraction. Method A that has a detection limit of 2 µg/g (uranium basis), is the preferred method because of its simplicity. However, if a lower detection limit is required, Method B should be used. Method B has a detection limit of 0.2 µg/g (uranium basis).

89.3 Boric acid is used in both Method A and Method B to form a complex with the fluoride prior to the extraction. A TBP-xylene mixture is used in both methods to extract the uranium, leaving the chromium in the aqueous phase, in which the final atomic absorption determination is made.

90. Interferences

90.1 In relatively pure UF_6, that is normally analyzed by this method, there is usually no problem with interferences.

91. Apparatus

91.1 *Atomic Absorption Spectrophotometer,* as described in Proposed Recommended Practices for Atomic Absorption Spectrometry.

92. Reagents

92.1 *Boric Acid* (H_3BO_3), reagent grade.

92.2 *Boric Acid Solutions,* saturated. Prepare by dissolving reagent-grade boric acid in deionized water until an excess of boric acid crystals remains undissolved.

92.3 *Chromium, Stock Solution, 1000 µg/mL*—Dissolve 3.7349 g of potassium chromate (K_2CrO_4) in 1 L of deionized water or use commercially available standard solutions.[11]

92.4 *n-Tributyl Phosphate,* purified.

92.5 *n-Tributyl Phosphate-Xylene Mixture (1 + 2)*—Mix 1 volume of TBP with 2 volumes of reagent-grade xylene.

92.6 *Xylene* (C_8H_{10}), reagent grade.

93. Procedure

93.1 *Method A:*

93.1.1 Hydrolyze the filtered UF_6 sample with chilled deionized water in accordance with Practice C1346.

93.1.2 Transfer an aliquot of the sample that contains 1 g of uranium to a 125-mL separation funnel.

93.1.3 Add 10 mL of saturated boric acid solution and enough concentrated HNO_3 to make the solution 2.5 *M* in HNO_3.

93.1.4 Add 25 mL of the (1 + 2) TBP-xylene mixture and shake for 30 s.

93.1.5 Allow the layers to separate, and transfer the bottom (aqueous) layer to a second 125-mL separation funnel.

93.1.6 Repeat 93.1.4 with the aqueous portion in the separation funnel.

93.1.7 Allow the layers to separate and collect the aqueous layer in a 50-mL volumetric flask.

93.1.8 Dilute to volume with water.

93.2 *Method B:*

93.2.1 Hydrolyze the filtered UF_6 sample with chilled deionized water in accordance with Practice C1346.

93.2.2 Weigh 3 g of boric acid into a 100-mL beaker.

93.2.3 Transfer an aliquot of the sample that contains 5 g of uranium to the beaker, and concentrate the mixture to less than 15 mL on a hot plate.

93.2.4 Add 10 mL of concentrated HNO_3, and transfer with minimum water to a 125-mL separation funnel.

93.2.5 Add 50 mL of the (1 + 2) TBP-xylene mixture and shake for 30 s.

93.2.6 Allow the layers to separate, and transfer the lower (aqueous) layer to a second 125-mL separation funnel.

93.2.7 Repeat 93.2.5 with the aqueous portion. Allow the layers to separate.

93.2.8 Transfer the lower (aqueous) layer to a 25-mL volumetric flask, and dilute to volume with water.

93.3 Analyze the aqueous extract by atomic absorption as follows:

93.3.1 Prepare the instrument for chromium analysis as outlined in the instrument manufacturer's atomic absorption manual.

93.3.2 Zero the instrument with deionized water.

93.3.3 Calibrate the instrument by preparing standards from UO_2F_2 solutions that have been spiked with known amounts of chromium. The standards are then extracted as outlined above. (Calibration is performed each time samples are analyzed.)

93.3.4 Analyze the sample extracts. Determine the concentration of chromium in the extracts from the calibration.

94. Precision and Bias

94.1 The relative standard deviation of a single analysis by this test method is approximately 10 % at the 2 µg/g concentration level, and the bias is + 1.5 % (relative). (A minimum of ten replicate measurements were used to determine the standard deviation and the bias of the test method.)

ATOMIC ABSORPTION DETERMINATION OF CHROMIUM INSOLUBLE IN URANIUM HEXAFLUORIDE

95. Scope

95.1 A test method is presented for the determination of chromium, insoluble in UF_6, by atomic absorption spectroscopy. The detection limit achieved using this technique is dependent upon the amount of sample filtered through the filter. A detection limit of 0.5µ g/g has been obtained using a 10-g sample.

96. Summary of Test Method

96.1 The liquid UF_6 sample is filtered through a porous nickel filter (see Practice C1689), and both the filter and the residue are dissolved in dilute nitric acid for the analysis. The amount of UF_6 filtered is determined by the detection limit required. The usual sample will vary between 10 and 50 g. The dissolved solution is diluted so as to contain a final nickel concentration of 1 % and an ammonium chloride concentration of 2 %. The chromium is then determined by atomic absorption spectrophotometry.

96.2 In the atomic absorption analysis, nickel suppresses the chromium response. To minimize this effect, it is necessary to add ammonium chloride. To further compensate for this and other matrix effects, it is necessary to prepare standard chromium solutions that contain both 1 % nickel and 2 % ammonium chloride. The concentration range of the standards prepared should bracket the expected concentrations in the samples.

97. Interferences

97.1 The interference of nickel and other metallic elements is controlled by the addition of ammonium chloride to suppress ionization.

98. Apparatus

98.1 *Atomic Absorption Spectrophotometer,* as described in Proposed Recommended Practices for Atomic Absorption Spectrometry.

99. Reagents

99.1 *Ammonium Chloride Solution, 200 g/L*—Dissolve 200 g of ammonium chloride, reagent grade, in deionized water and dilute to 1 L.

99.2 *Chromium Standard Solutions*—Prepare 100-mL quantities of a 1 % nickel solution containing 50, 30, 20, 10 and 5µg

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[11] Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219 is a suggested vendor.

of chromium. Add 10 mL of the ammonium chloride stock solution to each standard solution before diluting to volume.

99.3 *Chromium Stock Solution, 1000 µg/mL*—Dissolve 3.7349 g of potassium chromate (K_2CrO_4) in 1 L of deionized water or use commercially available standard solutions.

99.4 *Nickel Solution, 100 g/L*—Dissolve 100 g of spectrographically pure nickel powder in 500 mL of deionized water and 50 mL of concentrated HNO_3. Heat until dissolution is complete and dilute to 1 L with deionized water.

100. Procedure

100.1 Filter sufficient UF_6 to obtain the desired detection limit (see Practice C1689).

NOTE 17—The detection limit of the atomic absorption technique is 0.05 µg chromium/mL in a 1 % nickel solution. When a 1 g filter is dissolved and diluted to 100 mL, the weight of chromium needed in the determination to report a positive value is in excess of 5 µg. g. A 10-g uranium sample at this level would show 0.5 µg/g chromium, while a 50-g uranium sample would show 0.1 µg/g chromium.

100.2 Dissolve the filter and residue in a minimum amount of dilute HNO_3 (about 10 mL).

100.3 Transfer the solution to a 100-mL volumetric flask (for a 1-g filter), add 10 mL of the ammonium chloride solution, and dilute to volume with deionized water.

NOTE 18—For filters weighing other than 1 g, make appropriate dilution so that the final solution will contain 1 % nickel and 2 % ammonium chloride.

100.4 Analyze the diluted sample by atomic absorption as follows:

100.4.1 Prepare the instrument for chromium analysis as outlined in the instrument manufacturer's atomic absorption manual.

100.4.2 Zero the instrument with a blank 1 % nickel solution which contains 2 % ammonium chloride.

100.4.3 Determine the chromium by standard atomic absorption techniques, comparing the sample measurements to those of the prepared standards in the same concentration region.

101. Precision and Bias

101.1 The relative standard deviation of a single analysis using this test method is approximately 10 %. (Ten replicate measurements were made to determine the standard deviation of the procedure. A bias could not be computed, since no certified standard was available.)

DETERMINATION OF TECHNETIUM-99 IN URANIUM HEXAFLUORIDE

102. Scope

102.1 Technetium can be determined by ICP-MS (see Test Method C1287) or by liquid scintillation counting (see Test Method C1539). The following test method uses extraction and beta counting.

103. Summary of Test Method

103.1 The sample, to which a nonradioactive rhenium carrier has been added, is dissolved in HNO_3 and the solution is evaporated almost to dryness at low temperature (do not boil). A solution of potassium carbonate (K_2CO_3) and hydrogen peroxide (H_2O_2) is added, and the technetium is extracted into methyl ethyl ketone. The ketone is evaporated, and the residue of perrhenate and pertechnetate is dissolved in water. Stable cerium, cesium, ruthenium, and strontium are added to the residual solution to dilute any radioactive trace quantities of these elements that may precipitate with the ^{99}Tc. The technetium and rhenium are precipitated with tetraphenyl arsonium chloride, weighed in a tared stainless steel dish, and beta counted. The beta counting rate is corrected for the recovery indicated by the rhenium carrier, and compared to that of a ^{99}Tc standard.

104. Apparatus

104.1 *Automatic Low-level Beta Counter.*

104.2 *Counter Standard,* known activity of ^{90}Sr-^{90}Y, or some other well-characterized beta standard.

104.3 *Centrifuge.*

104.4 *Stainless Steel Dish,* 25 mm in diameter and 6.4 mm deep.

104.5 *Separation Funnel,* 250 mL.

105. Reagents

105.1 *Ammonium Hydroxide (NH_4OH),* concentrated.

105.2 *Boric Acid Solution,* saturated.

105.3 *Ferric Nitrate Solution*—Dissolve 18 g of $Fe(NO_3)_3 \cdot 9H_2O$ in distilled water and dilute to 250 mL.

105.4 *Hydrochloric Acid* (*HCl*), reagent grade, concentrated.

105.5 *Hydrogen Peroxide (H_2O_2),* 30 %.

105.6 *Methyl Ethyl Ketone,* reagent grade.

105.7 *Methyl Orange Indicator Solution.*

105.8 *Potassium Carbonate, 3 M*—Dissolve 414.6 g of K_2CO_3 in distilled water and dilute to 1 L.

105.9 *Potassium Perrhenate*—Dissolve 2 g of $KReO_4$ in distilled water and dilute to 1 L.

105.10 *Stable Solution of Cerium, Cesium, Ruthenium, and Strontium*—Dissolve 7.75 g of $Ce(NO_3)_3 \cdot 6H_2O$, 6.25 g of $RuCl_3$, 7.50 g of $SrCl_2 \cdot 6H_2O$, and 2.75 g of $CsNO_3$ in distilled water and dilute to 1 L.

105.11 *Technetium Standard in a Basic Aqueous Solution.*

105.12 *Tetraphenyl Arsonium Chloride (TPA)*— Dissolve 4 g of $(C_6H_5)_4AsCl$ in distilled water and dilute to 250 mL.

106. Procedure

106.1 *Sample Preparation:*

106.1.1 *Hydrolyzed UO_2F_2 Sample Preparation:*

106.1.1.1 Pipet 3 mL of $KReO_4$ solution into a beaker.

NOTE 19—**Caution:** The final precipitate of $(C_6H_5)_4AsReO_4$ must not exceed 14 mg to prevent shielding of ^{99}Tc.

106.1.1.2 Pipet 10 mL of the sample into the same beaker.

106.1.1.3 To complex the fluorides present, add 3 mL of saturated boric acid solution per gram of UO_2F_2.

106.1.1.4 Make basic by adding 10 mL of 3 *M* K_2CO_3 solution, and stir until the precipitate dissolves. (Uranyl hydroxide is soluble in K_2CO_3.) Add 2 mL of 30 % H_2O_2 to oxidize technetium to + 7. (Technetium must be present as Tc (+ 7) before extracting into ketone (**16**).)

106.1.2 *Control Sample Preparation:*

106.1.2.1 Pipet 3 mL of KR_eO_4 solution into a beaker.

NOTE 20—**Caution:** The final precipitate of $(C_6H_5)_4AsReO_4$ must not exceed 14 mg to prevent shielding of ^{99}Tc.

106.1.2.2 Pipet 10 mL of a ^{99}Tc solution of known activity level 100 dpm/mL into the same beaker.

106.1.2.3 Make basic by adding 10 mL of 3 *M* K_2CO_3 solution. Add 2 mL of 30 % H_2O_2 to oxidize the technetium to (+ 7) valence. (Technetium must be present as Tc (+ 7) before extracting into ketone.)

106.1.3 *Extraction:*

106.1.3.1 Transfer a sample in accordance with 106.1.1.1 and 106.1.2.1 to a separation funnel, wash the beaker in accordance with 106.1.1.1 and 106.1.2.1 with approximately 10 mL of 3 *M* K_2CO_3 , and drain the washings into the separation funnel.

106.1.3.2 Add approximately 50 mL of methyl ethyl ketone to the separation funnel and shake for about 2 min. Release the pressure after several seconds of agitation.

106.1.3.3 Drain the aqueous phase into a salvage container, and save the ketone that contains the rhenium and technetium.

106.1.3.4 Wash the sides of the separation funnel by shaking twice with 10-mL portions of 3 *M* K_2CO_3, and discard the washings.

106.1.3.5 Drain the ketone into a beaker.

106.1.3.6 Add 10 mL of distilled water to the same beaker in accordance with 106.1.3.5 and evaporate the ketone under heat lamps. (The water prevents the sample from going to dryness and the possible loss of Tc.)

106.1.4 *Precipitation:*

106.1.4.1 Transfer the solution to a 50-mL centrifuge tube. Wash the beaker with distilled water, and transfer the rinse solution to the tube.

106.1.4.2 Add three drops of concentrated HCl, five drops of the stable cerium, cesium, ruthenium, and strontium solution, and five drops of the ferric nitrate solution. Agitate slightly after each addition.

106.1.4.3 Precipitate $Fe(OH)_3$ by adding several drops of concentrated NH_4OH until the solution is basic to pH paper.

106.1.4.4 Filter the solution through a filter paper (for example, No. 41 Whatman) into another 50-mL centrifuge tube and discard the precipitate. (Technetium must be as Tc (+ 7) or else it will coprecipitate with the $Fe(OH)_3$.)

106.1.4.5 Add one drop of methyl orange indicator to the filtrate, and add HCl until the indicator turns red. Then add five extra drops of HCl.

106.1.4.6 Cool for 5 to 10 min in an ice bath, add 2 mL of TPA and stir (**16**).

106.1.4.7 Remove the solution from the ice bath, and centrifuge for 1 min at 3000 rpm.

106.1.4.8 Pour off the supernatant liquid. Wash the precipitate twice with approximately 10 mL of ice water.

106.1.4.9 Pour off the supernatant and dissolve the precipitate in 1 mL of acetone. Transfer the solution to a counting dish that has been weighed to ±0.1 mg.

106.1.4.10 Evaporate the precipitate to dryness under a heat lamp.

106.1.4.11 Weigh the dish, and calculate the net weight of the residual tetraphenyl arsonium perrhenate, $(C_6H_5)_4AsReO_4$.

106.1.4.12 Cover each dish with a layer of cellophane tape to prevent loss of the residue.

107. Counting

107.1 Beta count the counter standard until 30 000 counts are registered. Calculate the counting rate; it must be within ±2.5 % of the posted mean for the standard. (A typical value is 20 000 counts per minute (cpm).)

107.2 Beta count the sample and control sample.

107.3 Subtract the counter background (cpm) from both the sample and control sample counting rates to obtain the net cpm.

108. Calculations

108.1 Calculate rhenium carrier yield (*y*) for the sample and control sample as follows:

$$y = a/(b \times 2.19) \tag{10}$$

where:

a = net sample weight in beta counting dish (mg), and
b = net weight of $KReO_4$ added (mg).

(If 1 mg of $KReO_4$ is precipitated with tetraphenyl arsonium chloride, 2.19 mg of $(C_6H_5)_4AsReO_4$ is formed.)

108.2 Calculate the geometry factor (*GF*) as follows:

$$GF = d/c \tag{11}$$

where:

d = disintegrations per minute (dpm) of ^{99}Tc control sample, and
c = net cpm of control sample corrected for yield.

108.3 Calculate the ^{99}Tc in the sample as follows:

$$\mu g\ ^{99}\text{Tc/g sample} = (s \times GF)/(y \times 37\,807 \times g\ sample) \tag{12}$$

where:

s = net cpm of sample,
GF = geometry factor,
y = yield fraction of sample, and
37 807 = specific activity of ^{99}Tc in dpm/μg.

109. Precision and Bias

109.1 The relative standard deviation of an analysis is 25 % of the value in the range 0.01 to 0.05 μg ^{99}Tc/g sample. A minimum of ten replicate measurements was performed.

110. Sensitivity

110.1 The lower limit of detection is 0.001 μg ^{99}Tc with the Omni/Guard Low Background beta counter.

METHOD FOR THE DETERMINATION OF GAMMA-ENERGY EMISSION RATE FROM FISSION PRODUCTS IN URANIUM HEXAFLUORIDE

111. Scope

111.1 This method has been discontinued. The gamma activity of fission products can be determined using Test Method C1295. The beta activity of fission products can be determined according to Test Method C799 by beta counting after separation of uranium with TBP.

DETERMINATION OF METALLIC IMPURITIES BY ICP-AES

112. Scope

112.1 This test method is applicable to the determination of metallic impurities in uranium hexafluoride.

112.2 The elements determined are aluminum (Al), barium (Ba), beryllium (Be), bismuth (Bi), calcium (Ca), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), sodium (Na), nickel (Ni), lead (Pb), antimony (Sb), tin (Sn), vanadium (V), tungsten (W), zinc (Zn), and zirconium (Zr).

112.3 The detection limits range from 0.01 µg/gU to 0.6 µg/gU depending on the element.

112.4 This test method is also applicable to determination of metallic impurities in U_3O_8 and U-metal with no additional procedure changes.

113. Summary of Test Method

113.1 Uranium oxide, U_3O_8, generated in the pyrohydrolysis of uranium hexafluoride is dissolved in a solution of ammonium fluoride and nitric acid. The uranium is separated from the metallic impurities by solvent extraction using tri (2-ethylhexyl) phosphate diluted with heptane. The aqueous phase containing the metallic impurities is nebulized directly into the inductively coupled argon plasma and the metals are measured by emission spectroscopy (**17**, **18**).

114. Interferences

114.1 Uranium is a spectral interference and must be separated from the elements of interest. A uranium concentration of <100 µg/mL in the analyte solution can be tolerated.

115. Instrumentation and Apparatus

115.1 *Instrumentation:*

115.1.1 *Inductively Coupled Plasma-Emission Spectrometer*—Direct reading (simultaneous) or scanning (sequential) with a spectral bandpass of 0.05 nm or less. Either pneumatic or ultrasonic aerosol generators may be used to introduce the sample solution to the plasma source. An interactive computer based data and control system may be used.

115.1.2 The nebulizer and spray chamber should be resistant to hydrofluoric acid. These should be made of TFE fluorocarbon or polypropylene. A TFE-fluorocarbon-lined sample introduction tube with a sapphire tip is required in the torch assembly.

115.2 *Apparatus:*

115.2.1 *Peristaltic pump.*

115.2.2 *Beakers and lids,* TFE-fluorocarbon, 50 mL.

115.2.3 *Sample analysis tubes,* graduated, plastic, 50 mL.

115.2.4 *Separatory funnels,* TFE-fluorocarbon, 125 mL.

116. Reagents

116.1 *Argon*—High purity gas derived from the head space gas over the liquid in a liquid argon cylinder is recommended.

116.2 *Ammonium Fluoride (1 % W/V)*—Dissolve 1.0 g of ammonium fluoride in 100 mL of water. Store in a plastic bottle.

116.3 *Nitric Acid (sp. gr. 1.42)*—Concentrated nitric acid (HNO_3).

116.4 *Nitric Acid (8M)*—Add 1 volume of concentrated HNO_3 to an equal volume of water.

116.5 *Nitric Acid (1.6M)*—Add 1 volume of concentrated HNO_3 to 9 volumes of water.

116.6 *Tri(2-ethylhexyl) phosphate* $[(C_8H_{17}O)_3PO]$—Technical grade (TEHP).

116.7 *Heptane* $[CH_3(CH_2)_5CH_3]$.

116.8 *TEHP-Heptane Solution (1:1 V/V), purified*—Add 25 mL TEHP to a 250 mL separation funnel and dilute with 25 mL heptane. Add 75 mL of 8M HNO_3 to the funnel, stopper, and shake for 2 min. Discard the acid layer and retain the TEHP-heptane in the separation funnel. This purification step is carried out immediately before use.

116.9 *Calibration Solutions*—Prepare several compatible multi-element standard solutions in 4M nitric acid by adding appropriate volumes of single element stock solutions prepared by dissolving metals or metal salts of high purity. Commercial stock solutions may be used but should be verified. Calibration solutions with Zr should contain 1 % hydrofluoric acid by volume. Element concentration range of the solution should be 1 to 100 µg/mL.

NOTE 21—To avoid precipitation of alkaline earth fluorides, do not mix multielement standard solutions with Zr solutions. Compatibility of mixing commercial stock solution should be verified.

116.10 *Uranium Oxide Standards*—U_3O_8 standards containing metallic impurities.

116.11 *Uranium Oxide* (U_3O_8)—High purity to be used as a blank. See Appendix X3for preparation procedure.

117. Preparation of Apparatus

117.1 A set of suggested instrument operating parameters are given in Table 5. Operating parameters will vary with the type of instrument and the manufacturer's instructions should be followed.

117.2 A set of suggested analytical wavelengths is given in Table 6.

118. Calibration

118.1 *Calibration:*

118.1.1 Nebulize the multielement standard solutions, (see 116.9) to calibrate the instrument.

TABLE 5 Suggested Instrument Operating Parameters for ICP-AES Analysis

Argon Gas Flows	Coolant 21 L/min Plasma 0.6 L/min Nebulizer 0.4 L/min
RF Power	Forward 1100 W Reverse <10 W
Vertical Observation Zone	16 mm above the load coil
Integration Time	2 × 7 s on-line 1 × 7 s on-background
Nebulizer	Fixed cross flow Solution uptake 1 mL/min
Peristaltic Pump	Solution uptake 1.1 mL/min

TABLE 6 Suggested Wavelengths for ICP Analysis

Element	Wavelength, nm	Alternate Wavelength, nm
Al	308.2	...
Ba	455.4	493.4
Be	313.0	...
Bi	223.1	...
Ca	393.3	317.9
Cd	228.8	226.5
Co	228.6	...
Cr	205.6	267.7
Cu	324.8	...
Fe	259.9	...
Mg	279.6	280.3
Mn	257.6	...
Mo	202.0	204.6
Na	589.0	...
Nb	316.3	...
Ni	231.6	...
Pb	220.4	...
Sb	217.6	...
Sn	190.0	...
Ta	222.2	228.9
Ti	334.9	...
V	292.4	...
W	207.9	224.9
Zn	213.9	...
Zr	343.8	339.2

NOTE 22—The linear dynamic range should be established for each analytical channel and should cover the range expected in the samples.

118.1.2 Prepare calibration curves if automatic data processing equipment is not available.

119. Procedure

119.1 *Sample Preparation:*

119.1.1 Weigh a U_3O_8 sample containing 2 g of uranium to 0.001 g into a 50 mL TFE-fluorocarbon beaker.

NOTE 23—U_3O_8 sample from the pyrohydrolysis of UF_6 as described in section 17.

119.1.2 Add 10 mL of concentrated HNO_3 and 3 mL of 1 % NH_4F solution to the sample.

119.1.3 Cover the beaker with a TFE-fluorocarbon lid and heat the sample at 100°C for 10 min. After the sample dissolves, cool the solution.

119.1.4 Add 10 mL of water and mix the solution. Transfer the sample to a 125 mL separation funnel containing 50 mL of the purified TEHP-heptane solution. Wash the beaker with 1 to 2 mL of 8M HNO_3 and add the washings to the separation funnel.

119.1.5 Stopper the separation funnel and shake the funnel for 2 min. Allow the phases to separate.

119.1.6 Drain the aqueous phase into another 125 mL separation funnel containing 50 mL of purified TEHP-heptane. Stopper and shake for 2 min. Allow the phases to separate.

119.1.7 Drain the aqueous phase into a graduated analysis tube and dilute to 40 mL with water. Cap the tube and mix thoroughly.

119.1.8 Run a series of U_3O_8 standards and a U_3O_8 blank using the above procedure.

119.2 *Measurement:*

119.2.1 Nebulize the extracted uranium blank and standards to verify chemical preparation and extraction efficiency and to establish a reagent blank.

119.2.2 Nebulize extracted sample solutions from 119.1.7.

120. Calculation

120.1 If automatic data processing is not available to calculate the metal concentrations on a uranium basis the calculation is as follows:

$$\text{Metal, } \mu g/gU = A \times 40/(W \times 0.848) \tag{13}$$

where:

A = micrograms of metal per mL of the measured solution corrected for reagent blank,

W = weight of U_3O_8, in g.

121. Precision and Bias

121.1 *Precision*—The single operator and within laboratory precision of this method are shown in Table 7. Two precision estimates were made using the same U_3O_8 standard. The first estimate was based on eight separate determinations and the second estimate was based on five separate determinations.

121.2 *Bias*—A New Brunswick Laboratory reference material set (CRM No. 123, 18 Element Impurity Standard) was analyzed by this test method. Single analyses were made on CRM 123 (1-3) and duplicate analyses were made on CRM 123 (4-6). A comparison of the results to the reference values are shown in Table 8 and Table 9. Although the data are insufficient to establish an estimate of bias for the test method, the results compare very well with the reference values and fall within the range established by an inter-laboratory measurement program **(19)**.

TABLE 7 Analysis of Mallinckrodt U_3O_8—Standard A by ICP-AES

Element	µg Element/g U				
	Certified Value	Mean and Standard Deviation			
		Run 1		Run 2	
Al	64	64	8	64	2
Ba	61	61	3	63	2
Be	4.9	4.9	0.4	5.4	0.2
Bi	4.9	4.9	0.3	4.5	0.4
Ca	53	53	3	59	2
Cd	0.6	0.6	0.1	0.7	0.2
Cr	47	47	3	50	3
Cu	7.8	7.8	0.9	8.9	1
Fe	43	43	2	48	2
Mg	10	10	0.8	12	2
Mn	5	4.9	0.5	5.2	0.4
Ni	69	69	4	75	4
Sn	30	28	6	26	3
Zn	13	13	3	19	3

TABLE 8 Analysis of NBL-CRM 123, U_3O_8 by ICP-AES

Element	Standard, µg Element/gU					
	123-1		123-2		123-3	
	Ref.	Found	Ref.	Found	Ref.	Found
Al	205	200	105	110	55	65
B	5.1	4.8	2.6	2.5	1.1	1.0
Cd	5.2	4.5	2.7	2.3	1.2	0.9
Ca	200	213	100	113	51	58
Cr	102	114	52	60	22	25
Cu	50	50	25	26	10.4	11
Fe	210	206	110	114	60	60
Pb	51	50	26	26	10.8	11
Mg	101	100	51	60	21	26
Mo	100	95	50	48	20	19
Ni	202	211	102	110	52	54
Na	400	362	200	187	100	99
Sn	51	50	26	26	11	10
V	50	48	25	24	10	9
Zn	202	223	102	116	52	58

TABLE 9 Analysis of NBL-CRM 123, U_3O_8 by ICP-AES

Element	Standard, µg Element/gU					
	123-4		123-5		123-6	
	Ref.	Found	Ref.	Found	Ref.	Found
Al	24	28	14	17	9	9
B	0.7	0.6	0.4	0.3	0.3	0.4
Cd	0.5	0.4	0.25	0.2	0.1	0.25
Ca	20	28	10	15	5	8
Cr	15	14	10	8	7	5
Cu	5	5	2.5	2.8	1	1
Fe	28	30	18	20	13	14
Pb	5	5	2.5	3	1	1.3
Mg	10	14	5	8	2	4
Mn	5	6	2.5	2.9	1	1
Ni	20	22	10	12	5	6
Na	30	43	20	19	10	12
Sn	5	7	2.5	2.5	1	1.5
V	5	4	2.5	1.8	1	0.4
Zn	20	22	10	12	5	6
Zr	20	24	10	12	5	7

DETERMINATION OF MOLYBDENUM, NIOBIUM, TANTALUM, TITANIUM, AND TUNGSTEN BY ICP-AES

122. Scope

122.1 This test method is applicable to the determination of molybdenum, niobium, tantalum, titanium, and tungsten in uranium hexafluoride.

122.2 The limit of detection for each element is 0.1 µg/gU.

123. Summary of Test Method

123.1 Molybdenum, niobium, tantalum, titanium, and tungsten are separated from uranium by solvent extraction as their benzoylphenylhydroxylamine (BPHA) complexes into chloroform (9). The extract is evaporated to dryness and then wet ashed with nitric acid and sulfuric acid. The residue is ignited at 600°C and then dissolved in a mixture of hydrochloric and hydrofluoric acids and diluted with water. The solution is nebulized into an inductively coupled argon plasma and the metals are measured by emission spectroscopy (20, 17).

124. Interferences

124.1 Uranium is a spectral interference and must be separated from the elements of interest. A uranium concentration of <100 µg/mL in the analyte solution can be tolerated.

125. Instrumentation and Apparatus

125.1 *Instrumentation:*

125.1.1 *Inductively Coupled Plasma-Emission Spectrometer*—Direct reading (simultaneous) or scanning (sequential) with a spectral bandpass of 0.05 nm or less. Either pneumatic or ultrasonic aerosol generators may be used to introduce the sample solution to the plasma source. An interactive computer based data and control system may be used.

125.2 *Apparatus:*
125.2.1 *Peristaltic pump,*
125.2.2 *Platinum dishes with lids,* 75 and 250 mL
125.2.3 *Separatory funnels with TFE-fluorocarbon stopcocks,* 250 mL
125.2.4 *Beakers and lids,* TFE-fluorocarbon, 250 mL
125.2.5 *Graduates,* plastic, 50 mL
125.2.6 *Sample analysis tubes,* graduated, plastic, 50 mL
125.2.7 *Automatic shaker with clamps,* to hold 250 mL separation funnels.

126. Reagents

126.1 *Boric Acid Solution (10 %)*—Dissolve 100 g of boric acid, H_3BO_3, in 800 mL of hot water and dilute to one liter with water. Store this solution at 60°C.

126.2 *Ethyl Alcohol (C_2H_5OH),* absolute.

126.3 *N-Phenylbenzohydroxamic Acid Solution (2 %)*—Dissolve 20 g n-phenylbenzohydroxamic acid (BPHA) in 1 L of ethyl alcohol.

126.4 *Chloroform ($CHCl_3$).*

126.5 *Hydrochloric Acid (sp. gr. 1.19)*—Concentrated hydrochloric acid (HCl).

126.6 *Hydrofluoric Acid (48 %)*—Concentrated hydrofluoric acid (HF).

126.7 *Hydrofluoric Acid (2M)*—Add 83 mL of concentrated HF to 1 L with water. Store in a plastic bottle.

126.8 *Nitric Acid (sp. gr. 1.42)*—Concentrated nitric acid (HNO_3).

126.9 *Sulfuric Acid (sp. gr. 1.84)*—Concentrated sulfuric acid (H_2SO_4).

126.10 *Hydrochloric Acid—Hydrofluoric Acid Solution (1:5 V/V)*—Add one volume of concentrated hydrochloric acid (HCl) to five volumes of concentrated hydrofluoric acid (HF).

126.11 *Element Standard Stock Solutions (100 µg/mL)*—Dissolve 0.1000 g of high purity metal powder in 5 mL concentrated HF. Add concentrated HNO_3 dropwise to aid the dissolution. Dilute to 1.00 liter with 2*M* HF.

126.12 *Element Standard Solutions (10 µg/mL)*—Aliquot 100 mL of each standard stock solution into a one liter plastic volumetric flask and dilute to volume with 2*M* HF.

126.13 *Element Calibration Standards (0.1, 0.5, 1.0, and 5.0 µg/mL)*—Aliquot 1, 5, 10, and 50 mL of each element standard stock solutions into 4 one-liter plastic volumetric flasks. Add 25 mL of concentrated HNO_3 to each flask and dilute to volume with water.

126.14 *Uranium Standard Solution (100 g U/L)*—Dissolve 117.9 g of pure U_3O_8 with 100 mL of 8*M* HNO_3. Dilute to 1 L with water.

127. Preparation of Apparatus

127.1 A set of suggested instrument operating parameters are shown in Table 5. Operating parameters will vary with the type of instrument and the manufacturer's instructions should be followed.

127.2 A set of suggested analytical wavelengths are given in Table 6.

128. Calibration and Standardization

128.1 *Uranium Calibration Standards:*

128.1.1 Pipet two 50-mL aliquots of the 100 gU/L uranium standard solution into separate 250-mL TFE fluoro-carbon beakers.

128.1.2 Spike one uranium aliquot with 1 mL of the 10 µg/mL element standard solution, 146.12. The other uranium aliquot will be used as a blank.

128.1.3 Add 10 mL of concentrated HF to each solution.

128.1.4 Follow the procedure from 129.1.2 through 129.1.13.

128.2 *Calibration:*

128.2.1 Nebulize the multi-element standards 126.13 to calibrate the instrument.

NOTE 24—The linear dynamic range should be established for each analytical channel and should cover the range expected in the samples.

128.2.2 Prepare calibration curves if automatic data processing equipment is not available.

128.3 *Measurement:*

128.3.1 Nebulize the extracted uranium blank and standard to verify the chemical preparation, extraction and to establish a reagent blank.

128.3.2 Nebulize the extracted sample solutions.

129. Procedure

129.1 *Sample Preparation:*

129.1.1 Transfer an aliquot of UO_2F_2 solution containing 10 g of U to a 250 mL TFE fluorocarbon beaker.

NOTE 25—UF_6 sample hydrolyzed as described in Practices C1346 and C1689.

129.1.2 Add 50 mL of 10 % boric acid solution to the beaker and mix thoroughly.

129.1.3 Transfer the sample solution to a 250 mL separatory funnel.

129.1.4 Add 5 mL of the 2 % BPHA-ethyl alcohol solution and mix. Allow the solution to stand for 10 min.

129.1.5 Add 20 mL of chloroform, stopper, and shake for 10 min using an automatic shaker.

129.1.6 Allow the layers to separate and drain the chloroform layer into a 75 mL platinum dish.

129.1.7 Evaporate the chloroform extract to dryness on a hot plate at about 80°C.

NOTE 26—Direct a gentle stream of air over the surface to aid evaporation and keep the solution from creeping.

129.1.8 Add 2 to 3 mL of concentrated HNO_3 to the residue. Wait until the residue turns black.

129.1.9 Add 0.5 mL of concentrated H_2SO_4 and evaporate to dryness.

129.1.10 Cover the platinum dish with a lid and ignite the residue at 600°C for 1 h.

129.1.11 Dissolve the residue in 1 mL the HCl-HF mixture by heating the solution gently.

129.1.12 Transfer the solution to a 50 mL analysis tube.

129.1.13 Add 1 mL of concentrated HNO_3 and dilute to 40 mL with water.

130. Calculation

130.1 If automatic data processing is not available, calculate the metal concentrations on a uranium basis. The calculation is as follows:

$$\text{Metal } \mu\text{g/gU} = \frac{A \times 40}{W} \tag{14}$$

where:

A = micrograms of metal per mL of the measured solution corrected for reagent blank.

W = weight of U in UO_2F_2 aliquot (129.1.1).

131. Precision and Bias

131.1 *Precision*—The single operator and within laboratory precision of this method is shown in Table 10. These estimates are based on four separate analyses on uranyl fluoride solutions containing 10 g of U spiked at two levels.

131.2 *Bias*—Since there is no accepted reference material for determining bias in these test methods for measuring Mo, Nb, Ta, Ti, and W in UF_6, no statement of bias is being made.

132. Keywords

132.1 analytical methods; mass spectrometry; radiochemistry; uranium hexafluoride

TABLE 10 Precision Estimates for ICP-AES Analysis

Concentration	Relative Standard Deviation, %				
µ g/gU	Mo	Nb	Ta	Ti	W
1.0	6.0	7.3	6.2	9.3	7.4
2.0	3.4	5.4	4.4	3.4	5.6

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