



Designation: C1287 – 10

Standard Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry¹

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1. Scope

1.1 This test method covers the determination of 67 elements in uranium dioxide samples and nuclear grade uranium compounds and solutions without matrix separation by inductively coupled plasma mass spectrometry (ICP-MS). The elements are listed in Table 1. These elements can also be determined in uranyl nitrate hexahydrate (UNH), uranium hexafluoride (UF_6), triuranium octoxide (U_3O_8) and uranium trioxide (UO_3) if these compounds are treated and converted to the same uranium concentration solution.

1.2 The elements boron, sodium, silicon, phosphorus, potassium, calcium and iron can be determined using different techniques. The analyst's instrumentation will determine which procedure is chosen for the analysis.

1.3 The test method for technetium-99 is given in [Annex A1](#).

1.4 The values stated in SI units are to be regarded as standard.

1.5 *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.* **Warning**—The ICP-MS is a source of intense ultra-violet radiation from the radio frequency induced plasma. Protection from radio frequency radiation and UV radiation is provided by the instrument under normal operation.

2. Referenced Documents

2.1 ASTM Standards:²

¹ This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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² 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.

[C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder](#)

[C776 Specification for Sintered Uranium Dioxide Pellets](#)

[C787 Specification for Uranium Hexafluoride for Enrichment](#)

[C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals](#)

[C967 Specification for Uranium Ore Concentrate](#)

[C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ²³⁵U](#)

[C1346 Practice for Dissolution of \$UF_6\$ from P-10 Tubes](#)

[C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis](#)

[D1193 Specification for Reagent Water](#)

3. Summary of Test Method

3.1 The sample is dissolved in acid if it is not already a solution. A fixed quantity of internal standard is added to monitor and correct for signal instability. The level of impurities in the solution is measured by ICP-MS. Customized software calculates the concentration of each element.

3.2 Uranium-concentration-matched standard solutions are used to calibrate the ICP-MS instrument. The calibration is linear up to at least 0.2 $\mu\text{g/ml}$ (100 $\mu\text{g/g}$ U) for each analyte.^{3,4}

3.3 Microwave dissolution may be used as an alternate dissolution method.

4. Significance and Use

4.1 This test method is capable of measuring the elements listed in [Table 1](#), some of which are required by Specifications [C753](#), [C776](#), [C787](#), [C788](#), [C967](#) and [C996](#).

³ "ICP-MS Versus Conventional Methods for the Analysis of Trace Impurities in Nuclear Fuel," by Allenby, P., Clarkson, A. S., Makinson, P. R., presented at 2nd Surrey Conference on Plasma Source Mass Spectrometry, Guildford, UK, July 1987.

⁴ "Trace Metals in NBL Uranium Standard CRM 124 Using ICP-MS," by Aldridge, A. J., Clarkson, A. S., Makinson, P. R., Dawson, K. W., presented at 1st Durham International Conference on Plasma Source Mass Spectrometry, Durham, UK, September 1988.

TABLE 1 Reporting Limits of Impurity Elements

NOTE 1—The impurity elements were determined in 0.2 % uranium solutions, prepared following Section 8.

NOTE 2—Acquisition time = 10 s/isotope using peak jump mode.

NOTE 3—103 Rh was used as an internal standard. For the elements where the technique is identified as Perkin Elmer DRCII scandium was used as internal standard for boron, sodium and phosphorus. Rhodium was used as the internal standard for potassium, calcium and iron in Reaction Cell mode.

NOTE 4—The LRL is based on the within run standard deviation (S_p) of 20 uranium-matched blank determinations for each analyte. This limit equals $4 \times S_p$, rounded up to a preferred value in the series 1, 1.5, 2, 3, 4, 6, multiplied or divided by the appropriate integer power of ten.

NOTE 5—The upper reporting limit can be increased by extending the calibration to 10 µg/mL (5000 µg/g U) if the ICP-MS used has an extended dynamic range (EDR) accessory.

NOTE 6—For the elements where the technique is listed as P-E DRCII, the instrumentation may be specific to those elements. Alternatively cold plasma technique may be used and it is up to the analyst to perform testwork using spikes and reference materials and to determine the lower reporting levels. The impurity elements were determined in 0.16 % uranium solutions, prepared following Section 8. The dwell times are listed in 8.4.1.1.

NOTE 7—Some of the elements are not included in the material specifications and have been included only as a research record for the reader's interest.

Analyte	Mass Used	Analyte Group	Lower Reporting Limit (LRL), µg/g U	Upper Reporting Limit (URL), µg/g U	Technique
Lithium	7	A	0.01	100	normal plasma
Beryllium	9	A	0.04	100	normal plasma
Boron	11	E	0.3	100	DRCII
Sodium	23	E	0.3	100	DRCII
Magnesium	24	A	4	100	normal plasma
Aluminum	27	D	2	1000	normal plasma
Phosphorus	31	E	1	100	DRCII
Potassium	39	E	2.0	100	DRCII
Calcium	40	E	3	100	DRCII
Scandium	45	A	4	100	normal plasma
Titanium	48	B	0.2	100	normal plasma
Vanadium	51	B	0.04	100	normal plasma
Chromium	52	B	0.1	100	normal plasma
Manganese	55	A	0.1	100	normal plasma
Iron	56	A	0.2	100	DRCII
Cobalt	59	A	0.02	100	normal plasma
Nickel	60	A	0.4	100	normal plasma
Copper	65	A	0.2	100	normal plasma
Zinc	66	A	0.3	100	normal plasma
Gallium	69	A	0.04	100	normal plasma
Germanium	74	A	0.2	100	normal plasma
Arsenic	75	A	0.2	100	normal plasma
Selenium	82	A	3	100	normal plasma
Rubidium	85	A	0.06	100	normal plasma
Strontium	88	A	0.06	100	normal plasma
Yttrium	89	A	0.04	100	normal plasma
Zirconium	90	B	0.02	100	normal plasma
Niobium	93	B	0.01	100	normal plasma
Molybdenum	95	B	0.04	100	normal plasma
Ruthenium	102	B	0.02	100	normal plasma
Palladium	106	B	0.2	100	normal plasma
Silver	107	A	0.1	100	normal plasma
Cadmium	111	A	0.03	100	normal plasma
Indium	115	A	0.04	100	normal plasma
Tin	116	B	0.04	100	normal plasma
Antimony	121	B	0.02	100	normal plasma
Tellurium	130	B	0.4	100	normal plasma
Caesium	133	A	0.06	100	normal plasma

TABLE 1 Continued

Analyte	Mass Used	Analyte Group	Lower Reporting Limit (LRL), µg/g U	Upper Reporting Limit (URL), µg/g U	Technique
Barium	138	A	0.02	100	normal plasma
Lanthanum	139	C	0.1	100	normal plasma
Cerium	140	C	0.01	100	normal plasma
Praseodymium	141	C	0.01	100	normal plasma
Neodymium	146	C	0.01	100	normal plasma
Samarium	149	C	0.01	100	normal plasma
Europium	151	C	0.01	100	normal plasma
Gadolinium	158	C	0.01	100	normal plasma
Terbium	159	C	0.01	100	normal plasma
Dysprosium	163	C	0.01	100	normal plasma
Holmium	165	C	0.01	100	normal plasma
Erbium	166	C	0.01	100	normal plasma
Thulium	169	C	0.01	100	normal plasma
Ytterbium	174	C	0.01	100	normal plasma
Lutetium	175	C	0.01	100	normal plasma
Hafnium	178	B	0.01	100	normal plasma
Tantalum	181	B	0.01	100	normal plasma
Tungsten	184	B	0.01	100	normal plasma
Rhenium	187	A	0.02	100	normal plasma
Osmium	190	B	0.2	100	normal plasma
Iridium	193	B	0.2	100	normal plasma
Platinum	195	B	0.2	100	normal plasma
Gold	197	B	0.06	100	normal plasma
Mercury	202	A	0.4	100	normal plasma
Thallium	205	A	0.02	100	normal plasma
Lead	208	A	0.02	100	normal plasma
Bismuth	209	A	0.03	100	normal plasma
Thorium	232	B	0.01	100	normal plasma

5. Apparatus

5.1 ICP-MS, controlled by computer and fitted with the associated software and peripherals. May be fitted with cold plasma option. Current instrumentation is available with dynamic reaction cell or collision cell options.

5.2 Autosampler, with tube racks and disposable plastic sample tubes compatible with 5.1 (optional).

5.3 Variable Micropipettes:

- 5.3.1 10 µL to 100 µL capacity.
- 5.3.2 100 µL to 1000 µL capacity.
- 5.3.3 1000 µL to 10.00 mL capacity.

5.4 Volumetric Flasks:

- 5.4.1 50 mL capacity—polypropylene.
- 5.4.2 100 mL capacity—polypropylene.
- 5.4.3 1 L capacity—glass.

5.5 Platinum Dish—100 mL capacity.

5.6 Silica Beaker—250 mL capacity.

5.7 Watch Glasses—75 mm diameter.

5.8 Polypropylene Tubes—50 mL, with graduation marks and with caps.

6. Reagents

6.1 The sensitivity of the ICP-MS technique requires the use of ultra high purity reagents in order to be able to obtain the low levels of detection. All the reagents below are ultra high purity grade unless otherwise stated:

6.1.1 Element stock standards at 1000 µg/mL for all the elements in Table 1.

6.1.2 *Hydrofluoric Acid (HF)*, (40 g/100 g), 23 molar.

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

6.1.3 *Nitric Acid*—Concentrated nitric acid (HNO_3), 15 molar.

6.1.4 *Rhodium Stock Solution* (1000 $\mu\text{g/mL}$ Rh)—Commercially available solution (see [Note 1](#)).

NOTE 1—Rhodium stock solution is commercially available supplied with a certificate of analysis for the element and a full range of trace impurities. The solutions are prepared by the manufacturer using a variety of media designed to keep each element in solution for a minimum of one year.

6.1.5 *Sulfuric Acid*—Concentrated sulfuric acid (H_2SO_4), 18 molar.

6.1.6 *Uranium Standard Base Solution*—Uranyl nitrate solution to Specification [C788](#), of known uranium (100 g/L) and aluminum content ($\leq 2 \mu\text{g/g}$ U). The total metallic impurity (TMI) content must not exceed $50 \mu\text{g/g}$ U and no individual analyte must exceed $10 \mu\text{g/g}$ U.

6.1.7 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification [D1193](#), Type I.

6.1.8 *Ammonia*—Anhydrous, NH_3 , 99.9995 % minimum purity. Used with instruments fitted with dynamic reaction cell option.

7. Standards

7.1 Four separate mixed standard solutions (A, B, C, and E) are prepared to prevent the precipitation of some elements (as insoluble chlorides, fluorides etc; see [Table 1](#) for details of the analyte groups). Analyte group A contains element stock solutions prepared in HNO_3 or HNO_3/HF , analyte group B contains element stock solutions prepared in HCl or HCl/HF , analyte group C contains the rare earth element stock solutions, and analyte group E contains boron sodium silicon, phosphorus, potassium and calcium. The mixed standard solutions should be prepared to contain only the analytes of interest. Other combinations of mixed standard solutions may be prepared to minimize the precipitation of the analytes.

7.1.1 Mixed standard solution A is prepared from stock solutions of each element from analyte group A. Transfer 1000 μL of the stock solution (1000 $\mu\text{g/mL}$) of each element into a

50 mL polypropylene volumetric flask and add 500 μL of concentrated nitric acid. Dilute to 50 mL with water and mix. This multi-element standard contains 20 $\mu\text{g/mL}$ of each analyte in 1 % nitric acid. This solution must be used on the day of preparation.

7.1.2 Mixed standard solution B is prepared from stock solutions of each element from analyte group B. Transfer 1000 μL of the stock solution (1000 $\mu\text{g/mL}$) of each element into a 50 mL polypropylene volumetric flask and add 500 μL of concentrated nitric acid. Dilute to 50 mL with water and mix. This multi-element standard contains 20 $\mu\text{g/mL}$ of each analyte in 1 % nitric acid. This solution must be used within one week of preparation.

7.1.3 Mixed standard solution C is prepared from stock solutions of each element from analyte group C. Transfer 1000 μL of the stock solution (1000 $\mu\text{g/mL}$) of each element into a 50 mL polypropylene volumetric flask and add 500 μL of concentrated nitric acid. Dilute to 50 mL with water and mix. This multi-element standard contains 20 $\mu\text{g/mL}$ of each analyte in 1 % nitric acid. This solution must be used within one week of preparation.

7.2 Standard solution D is prepared from the stock solution of aluminum from analyte group D. Transfer 1000 μL of the stock solution (1000 $\mu\text{g/mL}$ Al) into a 50 mL polypropylene volumetric flask and add 500 μL of concentrated nitric acid. Dilute to 50 μL with water and mix. This standard contains 20 $\mu\text{g/mL}$ of aluminum in 1 % nitric acid. This solution must be used within one week of preparation.

7.3 Mixed standard solution E is prepared from stock solutions of each element from analyte group E. Transfer 1000 μL of the stock solution (1000 $\mu\text{g/mL}$) of each element into a 50 mL polypropylene volumetric flask and add 500 μL of concentrated nitric acid. Dilute to 50 mL with water and mix. This multi-element standard contains 20 $\mu\text{g/mL}$ of each analyte in 1 % nitric acid. This solution must be used within one week of preparation.

7.4 Rhodium internal standard solution is prepared from the stock solution. Transfer 1000 μL of the stock solution (1000 $\mu\text{g/mL}$ Rh) into a 100 mL polypropylene volumetric flask and add 1000 μL of concentrated nitric acid. Dilute to 100 mL with water and mix. This internal standard solution contains 10 $\mu\text{g/mL}$ Rh in a 1 % nitric acid solution. Other internal standards such as scandium may be used. With high mass elements the analyst may choose internal standards such as iridium or terbium. Other elements may be applicable as well but it is up to the analyst to conduct the appropriate testwork.

NOTE 2—Throughout this standard, references to Rh internal standard solution will include all other internal standard elements that may be used.

8. Procedure

NOTE 3—A uranium-free reagent blank is used to eliminate bias due to the analyte concentrations in the uranium standard base solution. A uranium-matched reagent blank is necessary to provide a constant acid concentration in the nebulized solution.

8.1 *Sample Preparation for the Determination of All Elements Except Boron:*

8.1.1 Weigh a portion of uranium oxide containing between 2.45 and 2.55 g of uranium into a platinum dish. Record the

weight to the nearest 0.001 g. For uranyl fluoride solutions prepared using Practice C1346 and uranyl nitrate solutions, aliquot between 2.45 and 2.55 g of uranium into a platinum dish. Use a variable volume plastic pipet for the transfer of uranyl fluoride solutions. Record the weight to the nearest 0.001 g.

8.1.2 Add 10 mL of water and 12.5 mL of concentrated nitric acid. Heat on a hotplate to assist dissolution.

8.1.3 Add 2.5 mL of hydrofluoric acid (40 g/100 g) and warm at about 80°C for 5 min.

8.1.4 Allow the solution to cool and transfer quantitatively to a 50 mL polypropylene volumetric flask. Dilute to 50 mL with water and mix. This solution contains 50 g of uranium per litre in 25 % nitric acid/5 % hydrofluoric acid.

8.1.5 Transfer 4.00 mL of the solution in 8.1.4 and 1.00 mL of the rhodium internal standard solution (see 7.4) into a 100 mL polypropylene volumetric flask. Dilute to 100 mL with water and mix. This solution contains 2 g of uranium per litre and 0.1 µg/mL Rh in 1 % nitric acid/0.2 % hydrofluoric acid.

8.1.6 A uranium-free reagent blank (see 8.3.1) and a control or recovery sample must be prepared with every run of samples.

8.1.7 Analyze these solutions as in 8.4 using the calibration solutions prepared in 8.3. The solutions must be analyzed within 8 h of preparation to minimize the effects of analyte precipitation.

8.2 Sample Preparation for the Determination of Boron:

8.2.1 Weigh a portion of uranium oxide, containing between 0.095 and 0.105 g of uranium into a graduated 50 mL polypropylene tube (or alternative). The accuracy of the graduations on the tube must be verified. Record the weight to the nearest 0.001 g. For uranyl fluoride solutions prepared using Practice C1346 and uranyl nitrate solutions, aliquot between 0.095 and 0.105 g of uranium using variable volume plastic pipets. Record the weight to the nearest 0.001 g.

8.2.2 Add 1 mL of water and 1.25 mL of concentrated nitric acid. Cap. Heat in a hot water bath at about 80°C to assist dissolution. Heat until all the sample is dissolved.

8.2.3 Cool to room temperature. Add 0.1 mL of hydrofluoric acid (40 g/100 g) and cap. Heat in a hot water bath at about 80°C for 5 min.

8.2.4 Allow the solution to cool. Add 0.5 mL of scandium internal standard solution (see 7.4). Dilute to 50 mL with water and mix. This solution contains 2 g of uranium per litre and 0.1 µg/mL Sc in 2.5 % nitric acid/0.2 % hydrofluoric acid.

8.2.5 A uranium-free reagent blank and a control or recovery sample must be prepared with every run of samples.

8.2.6 Analyze these solutions as in 8.4 using the calibration solutions prepared in 8.3. The solutions must be analyzed within 8 h of preparation to minimize the effects of analyte precipitation.

8.3 Preparation of Blanks and Calibration Standard Solutions:

8.3.1 For the Determination of All Elements Except Boron:

8.3.1.1 *Uranium-free Reagent Blank*—Transfer 12.5 mL of concentrated nitric acid and 2.5 mL of hydrofluoric acid (40 g/100 g) into a 50 mL polypropylene volumetric flask. Continue as instructed from 8.1.5 onwards.

8.3.1.2 *Uranium-matched Calibration Blank*—Transfer 2.00 mL of the uranium standard base solution (see 6.1.6; this is equivalent to 0.20 g of uranium) into a 100 mL polypropylene volumetric flask. Add 1000 µL of concentrated nitric acid, 200 µL of hydrofluoric acid (40 g/100 g) and 1000 µL of rhodium internal standard solution (see 7.4). Dilute to 100 mL with water and mix. This solution contains 2 g of uranium per litre and 0.1 µg/mL Rh in 1 % nitric acid/0.2 % hydrofluoric acid.

8.3.1.3 *Uranium-matched Calibration Standard*—Transfer 2.00 mL of the uranium standard base solution (see 6.1.6; this is equivalent to 0.20 g of uranium) into a 100 mL polypropylene volumetric flask. Add 1000 µL of concentrated nitric acid, 200 µL of hydrofluoric acid (40 g/100 g), 1000 µL of each mixed standard solution (see 7.1.1, 7.1.2 and 7.1.3) and 1000 µL of rhodium internal standard solution (see 7.4). Dilute to 100 mL with water and mix. This solution contains 2 g of uranium per litre, 0.2 µg/mL of each analyte (equivalent to 100 µg/g U) and 0.1 µg/mL Rh in 1 % nitric acid/0.2 % hydrofluoric acid.

8.3.2 For the Determination of Boron:

8.3.2.1 *Uranium-matched Reagent/Calibration Blank*—Transfer 2.00 mL of the uranium standard base solution (see 6.1.6; this is equivalent to 0.20 g of uranium) into a 100 mL polypropylene volumetric flask. Add 2.5 mL of concentrated nitric acid, 200 µL of hydrofluoric acid (40 g/100 g), and 1000 µL of scandium internal standard solution (see 7.4). Dilute to 100 mL with water and mix. This solution contains 2 g of uranium per litre and 0.1 µg/mL Sc in 2.5 % nitric acid/0.2 % hydrofluoric acid.

8.3.2.2 *Uranium-matched Calibration Standard*—Transfer 2.00 mL of the uranium standard base solution (see 6.1.7; this is equivalent to 0.20 g of uranium) into a 100 mL polypropylene volumetric flask. Add 2.5 mL of concentrated nitric acid, 200 µL of hydrofluoric acid (40 g/100 g), 1000 µL of mixed standard solution (see 7.1 and 7.3), and 1000 µL of scandium internal standard solution (see 7.4). Dilute to 100 mL with water and mix. This solution contains 2 g of uranium per litre, 0.2 µg/mL of each analyte (equivalent to 100 µg/g U) and 0.1 µg/mL Sc in 2.5 % nitric acid/0.2 % hydrofluoric acid.

8.4 Measurement of Elements by ICP-MS:

8.4.1 To avoid contamination problems when nebulizing the samples, which contain hydrofluoric acid, the nebulizer system (that is, spray chamber and nebulizer) must be made from fluorinated plastic materials (for example, TFE-fluorocarbon or polychlorotrifluoroethylene).

8.4.1.1 Set up the ICP-MS for the analysis using the parameters given in the manufacturer's operating manual. Nebulize the uranium-matched reagent/calibration blank solution to optimize conditions using the 103 Rh internal standard.

Example Instrument Operating Conditions

Solution Pumping Rate	Sample solution IN: 1.25 mL/min
ICP Incident Power	1400 watts
ICP Reflected Power	<10 watts
Plasma Argon Coolant	14 L/min at 70 psig
Plasma Argon Auxiliary	0.7 L/min at 70 psig
Plasma Argon Nebulizer	0.93 L/min at 40 psig
Integration Method	Valley Int.
Integration Area	0.80000 daltons (Atomic Mass Units—AMU)
Background Counts	10.0000 counts/s

Dead Time 100.000 μs
Safe Resting Mass 129.253 daltons (AMU)

8.4.1.2 Acquire the data for all blank, calibration standard, control/recovery and sample solutions using the ICP-MS for the analytes required using the masses specified by the element menu and given in Table 1. The element menu must also contain the mass for the internal standard (normally 103 Rh). Uranium-matched calibration solutions are run at the start and end of each run. Recalibration during the run may be necessary.

9. Calculation

9.1 The use of a uranium-free reagent blank allows the analyte concentrations in the uranium-matched blank to be ignored. For potassium, calcium and iron however, a uranium-matched reagent/calibration blank must be used. The concentration of potassium, calcium, and iron in the uranium standard base solution (Z) must be added to the sample concentration to avoid reporting biased results (that is, if the iron content of the uranium standard base solution is 2 μg/g U, the uranium-matched reagent/calibration blank contains 2 μg/g U and the uranium-matched calibration standard contains 102 μg/g U. The calculation software sets these values at zero and 100 μg/g U, respectively.)

NOTE 4—Analyte counts are normalized using the internal standard count ratio (ISCR).

$$ISCR = \frac{103 \text{ Rh counts for first solution nebulized}}{103 \text{ Rh counts for each subsequent solution}} \quad (1)$$

The first solution nebulized is usually the uranium-matched reagent/calibration blank solution. The normalization and calculation of analyte concentrations is performed by the ICP-MS software.

9.2 The element concentration, M (expressed as μg/g U), for the elements in 8.1 and 8.2 is calculated from:

$$M = \frac{(A_s - A_b)}{(A_c - A_u)} \times C_c \times 500 \quad (2)$$

for elements in 8.1, and

$$M = \left[\frac{(A_s - A_u)}{(A_c - A_u)} \times C_c \times 500 \right] + Z \quad (3)$$

for potassium, calcium, and iron.

where:

- C_c = the concentration of the element in the uranium-matched calibration standard solution (μg/mL),
- A_s = the normalized peak signal of each element in the sample,
- A_b = the normalized peak signal of each element in the uranium-free reagent blank solution,
- A_c = the normalized peak signal of each element in the uranium-matched calibration standard solution,
- A_u = the normalized peak signal of each element in the uranium-matched reagent/calibration blank solution, and
- Z = the potassium, calcium, and iron content of the uranium standard base solution (μg/g U).

TABLE 2 Precision Data Derived from PCS and CRM Samples

NOTE 1—Acquisition time = 10 s/isotope using peak jump mode. Acquisition time is 2.5 s / isotope for B, Na, P, K, Ca, Fe.

NOTE 2—Table 2 is a list of “between-run” standard deviations for a single determination based on the analysis of in-house primary control samples (PCS series), NBL Certified Reference Material CRM 124-2 and CRM 98-2.

NOTE 3—103 rhodium was used as the internal standard for all elements except scandium was used as the internal standard for B, Na, and P. Rhodium was used as the internal standard for potassium, calcium, and iron in Reaction Cell mode.

NOTE 4—Some of the elements are not included in the material specifications and have been included only as a research record for the reader’s interest.

Analyte	Isotope	Concentration, μg/g U	Standard Deviation, μg/g U	Number of Determinations
Lithium	7	A	A	...
Beryllium	9	10	1.5	10
Boron ^B	11	2.6	0.2	13
Sodium ^B	23	202	11	11
Magnesium ^B	24	52	3.7	5
Aluminum	27	21.5	2.5	50
Phosphorus ^C	31	198	8.7	12
Potassium ^C	39	267	10	8
Calcium ^B	40	104	7	14
Scandium	45	A	A	...
Titanium	48	2.0	0.21	29
Vanadium	51	2.0	0.19	27
Chromium	52	5.0	0.51	27
Manganese	55	5.0	0.80	10
Iron	56	107	5.3	11
Cobalt ^B	59	12.7	0.49	5
Nickel	60	22	3.2	7
Copper	65	25	4.6	6
Zinc ^B	66	101	3.5	5
Gallium	69	A	A	...
Germanium	74	A	A	...
Arsenic	75	1.0	0.14	10
Selenium	82	A	A	...
Rubidium	85	A	A	...
Strontium	88	N/A ^D
Yttrium	89	A	A	...
Zirconium	90	1.00	0.090	27
Niobium	93	1.00	0.095	15
Molybdenum	95	2.00	0.091	20
Ruthenium	102	2.00	0.141	17
Palladium	106	A	A	...
Silver	107	N/A
Cadmium	111	5.0	0.29	10
Indium	115	5.0	0.21	10
Tin	116	5.0	0.16	9
Antimony	121	1.0	0.10	27
Tellurium	130	A	A	...
Caesium	133	A	A	...
Barium	138	10	1.5	10
Lanthanum	139	A	A	...
Cerium	140	A	A	...
Praseodymium	141	A	A	...
Neodymium	146	A	A	...
Samarium	149	N/A
Europium	151	N/A
Gadolinium	158	N/A
Terbium	159	A	A	...
Dysprosium	163	N/A
Holmium	165	A	A	...
Erbium	166	A	A	...
Thulium	169	A	A	...
Ytterbium	174	A	A	...
Lutetium	175	A	A	...
Hafnium	178	1.00	0.093	35
Tantalum	181	1.00	0.100	27
Tungsten	184	1.00	0.060	27

TABLE 2 Continued

Analyte	Isotope	Concentration, µg/g U	Standard Deviation, µg/g U	Number of Determinations
Rhenium	187	A	A	...
Osmium	190	A	A	...
Iridium	193	A	A	...
Platinum	195	A	A	...
Gold	197	A	A	...
Mercury	202	A	A	...
Thallium	205	5.0	0.16	10
Lead	208	5.0	0.25	10
Bismuth	209	5.0	0.60	10
Thorium	232	5.00	0.020	22

^A The elements are not determined on a routine basis. Insufficient precision data are available but are expected to be similar to those of the analytes where data are available.

^B Data obtained from CRM 124-2.

^C Data obtained from CRM 98-2.

^D N/A = Data not available; still being obtained.

9.3 Corrections for isobaric effects are not needed when impurities are at or below the upper reporting limit. The isotopes listed in Table 1 can be measured without significant isobaric interference except 48-titanium which suffers an interference from 48-calcium. Four hundred µg Ca/g U is equivalent to about 1 µg Ti/g U. Other titanium isotopes (mass 47 or mass 49) can be used but the lower reporting limit is increased to 1.5 and 2 µg/g U, respectively.

9.4 There is a correction for the molecular interference of ArCl⁺ on arsenic at mass 75. This can be corrected by monitoring ArCl⁺ at mass 77 and correcting by direct proportion the contribution of ArCl⁺ at mass 75 from the known ratio of ArCl⁺ 77:75. This correction is only required if concentrations of chloride are greater than 10 µg/mL in the nebulized solutions.

10. Precision and Bias

10.1 Precision:

10.1.1 The primary control samples (PCS) used were prepared in-house. They were prepared by adding a known amount of each analyte, as a solution, to characterized, high purity UO₃ hydrate (UOH). The UOH was then dried, blended, and ignited to the octoxide (U₃O₈). The U₃O₈ was then thoroughly blended and checked for homogeneity by replicate analysis. To aid the validation of results, the standard solutions used to prepare the PCS materials were obtained from a different supplier than those used in this procedure.

10.1.2 The precision data obtained from the routine analysis of a PCS and CRM 124-2 is given in Table 2. The precision data was collected over a period of 18 months from the work of four analysts using one instrument.⁵ The precision data for

⁵ A VG PlasmaQuad PQ1, available from Fisons Instruments, Inc., 55 Cherry Hill Drive, Beverly, MA 01915, was used for this purpose.

the elements B, Na, P, K, Ca, and Fe was obtained from the analysis of CRM 124-2 and CRM 98-2. The data was collected over a period of more than 12 months from the work of 2 analysts using one instrument.⁶ The operating conditions in the following table were used to collect the data in the precision and bias tables.

Perkin Elmer DRCII Instrument Operating Conditions for Boron, Sodium, Phosphorus, Potassium, Calcium, and Iron	
Solution pumping rate	0.95 mL/min
ICP incident power	1150 W
Plasma argon coolant	15 L/min
Plasma argon auxiliary	1.2 L/min
Plasma argon nebulizer	1.0 L/min
Acquisition method	Peak jump
Number of points across mass peak	1
Dead time	35 nanoseconds
Dwell time/mass	Boron 2500 ms Sodium 2500 ms Phosphorus 2500 ms Potassium 2500 ms Calcium 2500 ms Iron 2500 ms Rhodium 2500 ms
Sweeps/reading	1
Readings/replicate	4
Sample cone	Platinum
Skimmer cone	Platinum
Reaction cell gas	Ammonia
Reaction cell gas flow	Potassium 1.2 mL/min Calcium 0.8 mL/min Iron 0.4 mL/min Rhodium 0.4 mL/min

10.2 Bias—Data to assess bias is given in Table 3, Table 4, Table 5. The data comes from the analysis of NBL CRM series U₃O₈. The second and third columns, labeled “Prepared Value” and “NBL Mean” provide estimates of the true amount of impurities in the standards.

10.2.1 The “Prepared Value” represents the expected calculated analyte level from the preparation process. The “NBL Mean” is the arithmetic mean of an interlaboratory measurement program designed by NBL for analytes measured. The data was published in 1984. The numbers in parentheses in the third column are explained in footnote D of Table 3 and Table 4. Because of the lack of agreement about the amount of impurities actually in the standards, a statement of bias is impossible to make. The data is provided to allow interested and knowledgeable readers to make their own assessment of the applicability of the test method to their circumstances.

11. Keywords

11.1 impurities; inductively coupled plasma—mass spectrometry; uranium; uranium dioxide; uranium hexafluoride; uranyl nitrate solutions; uranium oxide; uranium trioxide

⁶ The Perkin-Elmer Sciex DRCII is available from Perkin-Elmer Corp., 761 Main Avenue, Norwalk, CT 06859-0012.