INTERNATIONAL

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# Standard Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry[1]

This standard is issued under the fixed designation C1287; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method 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 *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 a specific warning statement, see Note 1.

NOTE 1—**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.

1.3 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.

1.4 The test method for technetium-99 is given in Annex A1.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 % $^{235}U$
C1346 Practice for Dissolution of $UF_6$ from P-10 Tubes
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 µg/ml (100 µg/g U) for each analyte.[3,4]

## 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.

## 5. Apparatus

5.1 *ICP-MS*, controlled by computer and fitted with the associated software and peripherals. May be fitted with cold plasma option.

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.

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[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.

[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 Elan 5000A P-E Elan 5000A scandium was used as internal standard.

NOTE 4—The LRL is based on the within run standard deviation ($S_b$) of 20 uranium-matched blank determinations for each analyte. This limit equals 4 × $S_b$, 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 Elan 5000A, 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.

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 | P-E Elan5000A |
| Sodium | 23 | E | 0.3 | 100 | P-E Elan5000A |
| Magnesium | 24 | A | 4 | 100 | normal plasma |
| Aluminum | 27 | D | 2 | 1000 | normal plasma |
| Silicon | 28 | E | 1.5 | 100 | P-E Elan5000A |
| Phosphorus | 31 | E | 1.5 | 100 | P-E Elan5000A |
| Potassium | 39 | E | 2 | 100 | P-E Elan5000A |
| Calcium | 44 | E | 6 | 100 | P-E Elan5000A |
| 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 | 15 | 100 | normal plasma |
| 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 |
| 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 |

5.3.3 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.

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

6.1.4 *Rhodium Stock Solution (1000 µg/mL Rh)*—Commercially available solution (see Note 2).

NOTE 2—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 (≤ 2 µg/g U). The total metallic impurity (TMI) content must not exceed 50 µg/g U and no individual analyte must exceed 10 µg/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.

**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 s / isotope for B, Na, Si, P, K, Ca ( mass 44).

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 45 scandium was used as the internal standard for B, Na, Si, P, K and Ca (mass 44).

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 | 2.9 | 0.3 | 8 |
| Sodium[B] | 23 | 206 | 10 | 8 |
| Magnesium[B] | 24 | 52 | 3.7 | 5 |
| Aluminum | 27 | 21.5 | 2.5 | 50 |
| Silicon[B] | 28 | 115 | 19 | 8 |
| Phosphorus[C] | 31 | 204 | 19 | 9 |
| Potassium[C] | 39 | 288 | 20 | 9 |
| Calcium[B] | 44 | 104 | 8 | 8 |
| 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 | [A] | [A] | ... |
| 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 |
| 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 analytes.
[C] Data obtained from CRM 98-2 analytes.
[D] N/A = Data not available; still being obtained.

## 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 µg/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 µg/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 µg/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 µg/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 µg/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 µg/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 µg/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 μg/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 μg/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 μg/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 μg/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 μg/mL Rh in a 1 % nitric acid solution. Other internal standards such as scandium (used with B, Na, Si, P, K and Ca) 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.

7.5 Diluent solution is prepared from rhodium stock standard solution. Transfer 1000 μL of the stock solution (1000 μg/mL Rh) into a 1 L volumetric flask and add 10.00 mL of concentrated nitric acid. Dilute to 1 L with water and mix. This diluent solution contains 1 μg/mL Rh in 1 % nitric acid solution. Other internal standard diluent solutions may be used.

NOTE 3—Throughout this standard, references to Rh internal standard solution will include all other internal standard elements that may be used.

## 8. Procedure

NOTE 4—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, Silicon, Potassium, and Calcium*:

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 and Silicon Potassium and Calcium*:

8.2.1 Weigh a portion of uranium dioxide, uranium octoxide or uranium trioxide 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.

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 Silicon Potassium and Calcium*:

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