



Standard Practice for Preparation and Dissolution of Uranium Materials for Analysis¹

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^{ε1} NOTE—Editorially relocated warning statement in 8.2.2 in June 2014.

1. Scope

1.1 This practice covers dissolution treatments for uranium materials that are applicable to the test methods used for characterizing these materials for uranium elemental, isotopic, and impurities determinations. Dissolution treatments for the major uranium materials assayed for uranium or analyzed for other components are listed.

1.2 The treatments, in order of presentation, are as follows:

Procedure Title	Section
Dissolution of Uranium Metal and Oxide with Nitric Acid	8.1
Dissolution of Uranium Oxides with Nitric Acid and Residue Treatment	8.2
Dissolution of Uranium-Aluminum Alloys in Hydrochloric Acid with Residue Treatment	8.3
Dissolution of Uranium Scrap and Ash by Leaching with Nitric Acid and Treatment of Residue by Carbonate Fusion	8.4
Dissolution of Refractory Uranium-Containing Material by Carbonate Fusion	8.5
Dissolution of Uranium—Aluminum Alloys Uranium Scrap and Ash, and Refractory Uranium-Containing Materials by Microwave Treatment	8.6

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.* Specific hazards statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*²

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C776 Specification for Sintered Uranium Dioxide Pellets

C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

D1193 Specification for Reagent Water

3. Summary of Practice

3.1 Many uranium-containing materials such as high-purity metals and oxides dissolve readily in various mineral acids. The dissolution of uranium-plutonium mixed oxides is covered in Practice **C1168**. Highly refractory materials require prior grinding of samples and fusions to affect even partial dissolution. Combinations of the mineral acid and fusion techniques are used for difficult to dissolve materials.^{3,4,5} Alternatively, the combination of acids and a high pressure microwave have been found to be effective with more difficult to dissolve materials and can also be used for materials which dissolve in mineral acid in place of heating with a steam bath or hot plate.

3.2 The dissolved materials are quantitatively transferred to tared polyethylene bottles for subsequent sample solution mass determination and factor calculation. Aliquants are obtained by mass for high-precision analysis or by volume for less precise analysis methods. Quantitative transfers of samples and subsequent solutions are required. The sample is rejected whenever a loss is incurred, or even suspected.

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 *Selected Measurement Methods for Plutonium and Uranium in the Nuclear Fuel Cycle*, Second Edition, C. J. Rodden, ed., U.S. Atomic Energy Commission, 1972.

⁴ *Analysis of Essential Nuclear Reactor Materials*, C. J. Rodden, ed., U.S. Atomic Energy Commission, 1964.

⁵ Larsen, R. P., "Dissolution of Uranium Metal and Its Alloys," *Analytical Chemistry*, Vol 31, No. 4, 1959, pp. 545–549.

¹ This practice 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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3.3 Solutions of dissolved samples are inspected for undissolved particles. Further treatment is necessary to attain complete solubility if particles are present. When analyzing the dissolved sample for trace impurities, caution should be exercised so the dissolution process does not cause the impurity to be lost or does not increase the level of impurity being determined significantly.

NOTE 1—The use of double distilled acids may be necessary for low level trace impurities. The use of plastic labware will be necessary so the dissolution does not increase the level of impurities being determined. This may be necessary in Section 8.6.

3.4 These dissolution procedures are written for the complete or nearly complete dissolution of samples to obtain destructive assay results on as near to 100 % of the sample as possible. When sample inhomogeneity is determined to be a major contributor to assay error, nondestructive assay (NDA) determinations on residues from the dissolution may be requested at an earlier stage than suggested in these procedures; the contribution of the error to the total assay may be propagated using the NDA assay value and errors for the residue, and it may be determined that the error contributed to the sample assay by the NDA determination on the residue is acceptable.

3.5 The accuracy of the analytical method should be considered when determining if complete dissolution of the sample is required for difficult to dissolve matrices.

4. Significance and Use

4.1 The materials covered that must meet ASTM specifications are uranium metal and uranium oxide.

4.2 Uranium materials are used as nuclear reactor fuel. For this use, these materials must meet certain criteria for uranium content, uranium-235 enrichment, and impurity content, as described in Specifications C753 and C776. The material is assayed for uranium to determine whether the content is as specified.

4.3 Uranium alloys, refractory uranium materials, and uranium containing scrap and ash are unique uranium materials for which the user must determine the applicability of this practice. In general, these unique uranium materials are dissolved with various acid mixtures or by fusion with various fluxes.

5. Apparatus

5.1 *Balances*, for determining the mass of samples and solutions.

5.2 *Sample Mixing Equipment*—Sample tumbler or mixer, as appropriate; riffle splitter, stainless steel.

5.3 *Furnace*—Muffle furnace, with fused silica tray to hold crucibles, capable of operation to 1200°C.

5.4 *Heating Equipment*—A steam bath in a hood; hot plates; infrared lamps; Bunsen and blast burner, with provision for

both gas and compressed air supply; microwave oven⁶ and high-pressure, heavy duty dissolution vessels.

5.5 *Hardware*—Metal weighing scoop; funnel racks; tongs; rubber policemen; tripods; silica triangles; board, heat dissipating, at least 6.35-mm (0.25-in.) thick.

5.6 *Beakers, Volumetric Flasks, and Bottles*—Borosilicate glass is generally recommended. However, the analyst should be sure that safety and sample contamination are considered when choosing appropriate containers. If the background levels of impurities such as boron, iron and sodium are being determined, then polypropylene or polytetrafluoroethylene containers and labware will be necessary in place of borosilicate glass.

5.7 *Glassware*—Borosilicate glass is generally recommended except as specified. Watch glasses or petri dishes, to cover beakers; funnels; stirring rods; crucibles, Vycor, with lids.

5.8 *Plasticware*—Wash bottle, polyethylene, 125-mL, for aliquanting; petri dishes; narrow mouth polyethylene bottles; plastic bottles, 60 mL; funnels, polypropylene; pipets, transfer.

5.9 *Volumetric Flask*—Polypropylene, 25 mL, 50 mL, and 100 mL.

5.10 *Pipettes 10 μ L—5 mL* (or equivalent). Accuracy of $\pm 3\%$ is adequate.

5.11 *Filter Paper*—Whatman Nos. 40 and 42, or equivalent.

5.12 *Filter Paper Pulp*.

5.13 *Platinum Ware*—Crucibles, with lids; platinum-tipped tongs; dishes, with lids.

5.14 *TFE Fluorocarbon Ware*—Stirring rods.

5.15 *Dry Atmosphere Box*.

5.16 *Drying Oven*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade or better chemicals shall be used in all tests; impurities analyses, for example, may require that all reagents and standards be prepared using Plasma grade, trace metal grade (TMG), double distilled, or better. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where

⁶ The sole source of supply of the apparatus known to the committee at this time is CEM Corporation, 3100 Smith Farm Road, Mathews, NC 28105. If you are aware of alternative suppliers, please provide the information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of measurements made on the prepared materials.

6.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean laboratory-accepted demineralized or deionized water. For impurities analyses, Type 1 Reagent Grade⁸ water may be required dependent upon the accuracy and precision of the analysis method used.

6.3 Nitric Acid (HNO₃), concentrated (sp gr 1.4), 16 M.

6.4 HNO₃, 8 M—Add 500 mL of concentrated HNO₃ (sp gr 1.4) to approximately 400 mL of water and dilute to 1 L.

6.5 HNO₃, 10 % Add 100 mL of concentrated HNO₃ (sp gr 1.4) to 800 mL. Type 1 Reagent Grade water and dilute to 1 L.

6.6 HNO₃, 2 % Add 20 mL of concentrated HNO₃ to 900 mL. Type 1 Reagent Grade water and dilute to 1 L.

6.7 Hydrochloric Acid (HCl), concentrated 12 M (sp gr 1.2).

6.8 Hydrofluoric Acid (HF), concentrated 29 M (sp gr 1.2).

6.9 HF 7.2 M Add 250 mL of concentrated HF, Electronic Grade (29M), to 700 mL Type 1 Reagent Grade water and dilute to 1 L.

6.10 Sulfuric Acid (H₂SO₄), concentrated 18 M (sp gr 1.8).

6.11 Sulfuric Acid, 9 M—Add 500 mL of concentrated (sp gr 1.8) H₂SO₄ to approximately 400 mL of water, cool and dilute to 1 L. Store in a glass bottle.

6.12 Sodium Carbonate (Na₂CO₃).

6.13 Sodium Bisulfate (NaHSO₄).

7. Hazards

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

7.2 Review the material safety data sheets and safety procedures in the laboratory's safety manual before performing this procedure.

7.3 Elemental uranium is very reactive; assure initial reactions have subsided before sealing closed vessels. As turnings and powder, uranium is extremely pyrophoric, often igniting as a result of mechanical friction, a small addition of acid or water, or even spontaneously. The reaction of uranium alloys with acids may create an explosive mixture.³

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

⁸ See Specification D1193.

7.4 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.

8. Procedures

8.1 Dissolution of Uranium Metal and Oxide with Nitric Acid:

8.1.1 Clean the surface oxide from metallic uranium by placing the metal in a small beaker and adding enough 8 M HNO₃ to cover it. Place the beaker on a steam bath for 10 to 20 min to remove the surface oxide. When the black oxide has been removed completely, decant the supernatant liquid into the appropriate container, and rinse the metal twice with distilled water into the container.

8.1.1.1 Dry the metal by rinsing twice with acetone or ethanol. Place the metal on filter paper, and allow it to dry for 30 to 60 s, rolling the metal several times to expose all faces to the atmosphere.

8.1.1.2 Tare a weighing scoop on an analytical balance. Place the dry uranium metal from 8.1.1.1 in the scoop and weigh. Record the mass of the uranium metal (12 g of metal will provide approximately 2 L of 6 g/L solution; the ratios of metal mass and solution mass may be adjusted, as needed, to provide the desired concentration).

NOTE 2—Measure and record the room temperature, barometric pressure, and percent relative humidity if performing buoyancy corrections.

8.1.2 Tare a 2-L flask or polyethylene bottle on a top loader balance, or record the mass of the flask or bottle.

8.1.3 Transfer the metal quantitatively to the tared (or weighed) flask or bottle.

8.1.4 Add 250 mL of 8 M HNO₃ (adjust the nitric acid volume in ratio to the metal to be dissolved since insufficient HNO₃ will cause the metal surface to become passive) to the flask or bottle. Warm the flask or bottle on a steam bath (the flask or bottle must be left unstoppered due to gas generation, but it may be covered by an inverted beaker).

NOTE 3—If desired, up to 20 mL of concentrated H₂SO₄ may be added to the mixture. This will speed dissolution and ease later dissolution of the aliquants.

8.1.5 When the dissolution is complete, remove the flask or bottle from the steam bath, and allow it to cool to ambient temperature for ease of handling.

8.1.6 Dilute the solution to approximately 1900 mL by adding distilled water in 200 to 300-mL portions and swirling after each addition. Allow the solution to cool to room temperature, dilute to 2 L, and add a stopper or top. **Warning**—Do not invert the flask or bottle prior to obtaining the mass of the solution.

8.1.7 Weigh the full flask or bottle using the top-loader balance, and record the solution weight.

8.1.8 Invert the flask or bottle several times to mix the contents thoroughly prior to preparing aliquants.

8.2 *Dissolution of Uranium Oxides with Nitric Acid and Residue Treatment*—Common laboratory techniques are described in **Annex A1**. The techniques are referenced to the appropriate section in parentheses at the first place in the procedure where they may be applicable.

8.2.1 *Sample Preparation*—Obtain the mass of the sample using a four-place balance (usually 0.5-g to 0.1-mg sensitivity). Transfer the sample quantitatively to a beaker (**A1.1.1**). If the sample is a powder, cover it gently with distilled water. Cover the beaker with a watch glass. **Warning**—Do not wash down the walls of the beaker because the powder may creep up the sides of the beaker and be lost.

8.2.2 *Acid Dissolution* (**Warning**—Do not wet the beaker walls with the acid.):

8.2.2.1 Add approximately 100 mL of 8 M HNO₃ to the sample carefully in order to control the reaction rate. **Warning**—Powders may react very rapidly. If the reaction is too rapid, add distilled water to decrease the reaction rate.

8.2.2.2 Allow the reaction to subside; then heat on a steambath or hot plate (**A1.1.2**). Add additional 8 M HNO₃ as necessary, until dissolution is complete.

8.2.2.3 When the dissolution appears to be complete, wash down the walls of the beaker with distilled water and heat for an additional 30 min.

8.2.2.4 Allow the solution to cool; then filter (**A1.1.3 – A1.1.6**) into a beaker.

8.2.2.5 Place the filter paper in a platinum crucible (**A1.1.7**). Dry the filter paper(s) in the platinum crucible by placing it in a cold muffle furnace that is then set to 700°C; maintain the muffle furnace temperature at 700°C for at least 1 h for ignition of the crucible or dish contents, or until no carbon is visible.

8.2.2.6 Allow the crucible to cool; then add approximately 5 mL of concentrated HNO₃, 5 to 10 drops of HF, and 1 to 2 drops of 9 M H₂SO₄, and fill to near the top with distilled water. Heat to fumes of SO₃ on a hot plate.

8.2.2.7 Cool and add 2 mL of distilled water and 1 mL of concentrated HNO₃.

8.2.2.8 If the solution is clear, transfer it to the beaker containing the filtrate. Proceed to **8.2.5**.

8.2.2.9 If the solution is cloudy or contains solids, evaporate it to dryness. Proceed to **8.2.3** unless there is significant residue. Proceed to **8.2.4** if significant residue is present.

NOTE 4—Platinum crucibles are attacked slightly during a sodium bisulfate fusion. The fusion can be performed without the introduction of significant amounts of platinum into the sample only if the amount of residue is small. Perform a sodium carbonate fusion if significant residue is present.

8.2.3 *Sodium Bisulfate Fusion:*

8.2.3.1 Add approximately 0.5 g of NaHSO₄ to the crucible.

8.2.3.2 Holding the crucible with platinum-tipped tongs, heat the crucible carefully and slowly with a flame from a Bunsen or blast burner until the flux melts and clears.

8.2.3.3 Remove the crucible from the flame and allow it to cool.

8.2.3.4 Dissolve the fusion cake in the crucible in distilled water, and transfer the solution and any undissolved material to a beaker.

8.2.3.5 Add approximately 2 mL of concentrated HNO₃ to the beaker.

8.2.3.6 Cover the beaker with a watch glass, and heat on a steam bath until any remaining salts dissolve completely.

8.2.3.7 Remove the beaker from the steam bath and allow the solution to cool.

8.2.3.8 If the solution is clear, transfer it with distilled water to the beaker containing the filtrate. Proceed to **8.2.5**.

8.2.3.9 If the solution is cloudy or contains solids, filter it into the beaker containing the filtrate.

8.2.3.10 Place the filter paper in a platinum crucible. Dry the filter paper(s) in the platinum crucible by placing it in a cold muffle furnace that is then set to 700°C; maintain the muffle furnace temperature at 700°C for at least 1 h for ignition of the crucible or dish contents, or until no carbon is visible. If the ignition does not remove carbon completely, digest the sample with several drops of concentrated H₂SO₄ and fume to dryness on a hot plate. **Warning**—The sample may foam during the next fusion if carbon is not removed.

8.2.3.11 Allow the crucible to cool, and then proceed to **8.2.4**.

8.2.4 *Sodium Carbonate Fusion:*

8.2.4.1 Add a volume of Na₂CO₃ (1 to 4 g) to the platinum crucible that is approximately ten times the volume of residue. Cover the crucible with a platinum lid, and place it on a triangle supported by a tripod in a hood. Heat the crucible carefully with a flame from a bunsen or blast burner until the flux melts; then increase the air supply to attain maximum temperature. Alternatively, if proper safety precautions are followed, the crucible may be heated in a muffle furnace set initially at 300°C and then increased to 900°C.

8.2.4.2 Using platinum-tipped tongs, remove the lid and carefully swirl the crucible contents to mix. Replace the lid and heat the crucible in the flame for 5 to 10 min. Remove the crucible from the flame and allow it to cool.

8.2.4.3 Proceed to **8.2.4.5** if the melt is clear.

8.2.4.4 If undissolved material is visible in the melt, add 1 to 2 g of additional Na₂CO₃, warm the melt, repeat the step given in **8.2.4.2** once, and then proceed to **8.2.4.5**.

8.2.4.5 Place the crucible and lid in a beaker and cover with distilled water.

8.2.4.6 Add concentrated HNO₃ slowly and carefully until the reaction stops, covering the beaker with a watch glass after each addition of acid. Remove the crucible and lid from the beaker, using 8 M HNO₃ to rinse. Add 10 mL of concentrated HNO₃, and heat the covered beaker on a steam bath for 1 to 2 h.

8.2.4.7 Remove the beaker from the steam bath and allow the solution to cool.

8.2.4.8 If the solution is clear, transfer it to the beaker containing the filtrate. Proceed to 8.2.5.

8.2.4.9 If the solution is cloudy or contains solids, filter it into the beaker containing the filtrate.

8.2.4.10 Place the filter paper in a platinum crucible. Dry the filter paper in the platinum crucible by placing it in a cold muffle furnace that is then set to 700°C; maintain the muffle furnace temperature at 700°C for at least 1 h for ignition of the crucible or dish contents, or until no carbon is visible.

8.2.4.11 Allow the crucible to cool, then dry transfer as much of the residue as possible to a plastic petri dish, and submit it for uranium assay. Save the crucible, which may contain a small amount of residue, until it is determined whether further treatment is required.

8.2.4.12 If the residue contains less than 0.01 % of the amount of uranium estimated to be in the sample, proceed to 8.2.5.

8.2.4.13 If the residue contains more than 0.01 % of the amount of uranium estimated to be in the sample, the fusion must be repeated or alternate methods from references must be used.

8.2.5 Final Sample Solution Preparation:

8.2.5.1 Obtain the mass of an appropriately sized polyethylene bottle on a top loader balance. Record the bottle mass.

8.2.5.2 Transfer the sample to the bottle using a funnel. If more than one bottle is needed to contain the filtrate, a composite of the individual bottles must be prepared.

8.2.5.3 Calculate the desired final mass of the solution that will result in the desired concentration of uranium (usually approximately 10 mg of uranium per gram of solution).

8.2.5.4 Add enough distilled water to the solution to bring it to approximately the desired mass.

8.2.5.5 Weigh the bottle containing the solution on the same top loader balance. Record the weight of the bottle plus solution.

8.2.5.6 Invert the bottle to mix, and calculate the dilution factor.

NOTE 5—Remove aliquants of the solution for analysis as quickly as possible after the dissolution is complete; there is a danger of the material precipitating out of the solution with time.

8.3 Dissolution of Uranium-Aluminum Alloys in Hydrochloric Acid with Residue Treatment—Common dissolution techniques are described in Annex A1. The techniques are referenced to the appropriate section by a superscript at the first place in the procedure where they may be applicable.

8.3.1 Sample Preparation—Obtain the mass of the sample using a four-place balance (usually 5 to 15-g to 0.1-mg sensitivity). Transfer the sample quantitatively to a beaker (A1.1.1). Washing down the walls of the beaker, add enough distilled water to cover the sample at least 12.7-mm (0.5-in.) deep. Cover the beaker with a watch glass.

8.3.2 Acid Dissolution—Lift the watch glass and carefully add concentrated HCl dropwise to the sample beaker until no reaction is observed upon addition. If the reaction becomes too violent (spitting or foaming vigorously), stop, and add distilled water. Resume the acid addition when the reaction subsides.

8.3.2.1 Cover and heat the beaker on a hot plate set at a medium temperature until the solution is hot.

8.3.2.2 While stirring with a glass rod, add concentrated HNO₃ very slowly and carefully, until the solution turns from green to yellow.

8.3.2.3 Add an additional 25 mL of concentrated HNO₃, wash down the walls of the beaker with distilled water, cover, and place the sample beaker on a steambath (A1.1.2) to reflux until dissolution is complete.

8.3.2.4 Remove the beaker from the steambath and allow the solution to cool.

8.3.2.5 If the solution appears clear, filter (A1.1.3 – A1.1.6) it into a beaker using Whatman No. 42 filter paper, or equivalent.

8.3.2.6 If the solution is cloudy or murky, filter it first into a beaker using Whatman No. 40 filter paper, or equivalent. Then refilter the solution into another beaker using Whatman No. 42 filter paper, or equivalent.

8.3.2.7 Rinse the filter paper(s) thoroughly with distilled water until no trace of yellow color (uranium) remains.

NOTE 6—Leaving residual amounts of HCl and HNO₃ in the filter paper(s) will result in attack of the platinum crucible or dish in which the filter paper will be ignited.

8.3.2.8 Place the filter paper(s) in a platinum crucible or dish (A1.1.7). Dry the filter paper(s) in the platinum crucible or dish by placing it in a cold muffle furnace that is then set to 700°C; maintain the muffle furnace temperature at 700°C; for at least 1 h for ignition of the crucible or dish contents, or until no carbon is visible.

8.3.2.9 Allow the platinum crucible or dish to cool (A1.1.8); then add approximately 5 mL of concentrated HNO₃ and 5 to 10 drops of concentrated HF. Fill the crucible or dish with distilled water. Evaporate to dryness on a hot plate to remove silicon.

8.3.2.10 Repeat the step given in 8.3.2.9, as necessary, until no further reduction in the amount of residue is observed.

8.3.2.11 Cool and add 2 mL of distilled water and 1 mL of concentrated nitric acid.

8.3.2.12 If the solution is clear, add it to the filtrate; then proceed as in 8.2.5.

8.3.2.13 If the solution is not clear, evaporate it to dryness, and proceed as in 8.3.3.

8.3.3 Sodium Carbonate Fusion:

8.3.3.1 Add a volume of Na₂CO₃ (1 to 10 g) to the platinum crucible that is approximately 10 times the volume of residue. Cover the crucible with a platinum lid, and place it on a triangle supported by a tripod in a hood. Heat the crucible carefully with a flame from a bunsen or blast burner until the flux melts; then increase the air supply to attain maximum temperature. Alternatively, if proper safety precautions are followed, the crucible may be heated in a muffle furnace set initially at 300°C and then increased to 900°C.

8.3.3.2 Using platinum-tipped tongs, remove the lid and carefully swirl the crucible contents to mix. Replace the lid, and heat the crucible in the flame for 4 h.

8.3.3.3 Remove the crucible or dish from the flame, swirl until the flux solidifies, and allow it to cool.

8.3.3.4 Place the crucible or dish and lid in a beaker, and cover it with distilled water. Add concentrated HNO₃ slowly and carefully until the reaction stops, covering the beaker with

a watch glass after each addition of acid. Remove and rinse the crucible or dish and lid into the beaker with 8 M HNO₃.

8.3.3.5 Add 10 mL of concentrated HNO₃, and heat the covered beaker on a steam bath for 1 to 2 h. Remove the beaker from the steam bath and allow the solution to cool.

8.3.3.6 If the solution is clear, transfer it to the beaker containing the filtrate. Proceed as in 8.2.5.

8.3.3.7 If the solution is cloudy or contains solids, filter it into the beaker containing the filtrate.

8.3.3.8 Place the filter paper in a platinum crucible or dish. Dry the filter paper in the platinum crucible or dish by placing it in a cold muffle furnace that is then set to 700°C; maintain the muffle furnace temperature at 700°C for at least 1 h for ignition of the crucible or dish contents, or until no carbon is visible.

8.3.3.9 Allow the crucible or dish to cool to ambient temperature; then add 5 mL of 8 M HNO₃ and heat on a steam bath to dissolve.

8.3.3.10 If the solution is clear, add it to the filtrate. Proceed as in 8.2.5.

8.3.3.11 If the solution is not clear, evaporate it to dryness, and repeat the steps described in 8.3.3.1 – 8.3.3.9 once more; then proceed as in 8.2.4.11 through 8.2.4.13.

8.4 *Dissolution of Uranium Scrap and Ash by Leaching with Nitric Acid and Treatment of Residue by Carbonate Fusion*—Common dissolution techniques are described in Annex A1. The techniques are referenced to the appropriate section by a superscript at the first place in the procedure where they may be applicable.

8.4.1 *Sample Preparation for Small Samples and Large Acid-Soluble Samples*—If the sample is larger than 10 g and is not acid soluble, proceed to 8.4.2. If the sample is less than 10 g, the entire sample is leached with nitric acid. The entire sample is also leached when the samples are larger than 10 g and a significant portion of the sample matrix will dissolve during the acid leach. Obtain the mass of the sample using an analytical balance (usually with 0.1-mg sensitivity). Transfer the sample quantitatively to the appropriate container.

8.4.1.1 If the sample appears to contain significant amounts of silica or carbonaceous material, it should be poured into a platinum dish or crucible for treatment before HNO₃ leaching.

8.4.1.2 If the sample does not appear to contain significant amounts of silica or carbonaceous material, the sample should be poured into a beaker (A1.1.1).

8.4.1.3 Cover the container with a watch glass or platinum lid, as appropriate.

8.4.1.4 Proceed to 8.4.3 if the sample contains a large amount of carbon.

8.4.1.5 Proceed to 8.4.4 if the sample contains a large amount of silicon but does not appear to contain carbon.

8.4.1.6 Proceed to 8.4.5 if the sample does not contain significant amounts of either carbon or silica.

8.4.2 *Sample Preparation for Large Acid-Insoluble Samples*—Samples larger than 10 g, which are not significantly acid soluble, may be subjected to appropriate grinding, mixing, and splitting operations to obtain 5 to 10-g portions that are representative of the entire sample.

8.4.2.1 Perform appropriate grinding, mixing, and splitting operations to prepare duplicate 5 to 10-g portions of the sample in separate bottles. Samples that are hygroscopic may need to be prepared in a dry box.

8.4.2.2 Perform the step given in 8.4.1 for each duplicate.

8.4.3 *Samples Containing Carbonaceous Material*—Place the crucible or dish containing the sample in a muffle furnace. Remove the lid and ignite (A1.1.7) at 700°C for 1 to 2 h, or until no carbon is visible. Allow the crucible or dish to cool (A1.1.8) to ambient temperature.

8.4.3.1 Proceed to 8.4.4 if the sample contains significant amounts of silica.

8.4.3.2 Proceed to 8.4.3.3 if the sample does not contain significant amounts of silica.

8.4.3.3 Transfer the sample with water to a 600-mL beaker. If necessary, 8 M HNO₃ may be used to complete the transfer. Cover the beaker with a watch glass. Proceed to 8.4.5.

8.4.4 *Samples containing Large Amounts of Silica*—Remove the lid, cover the sample gently with water, and place the platinum dish on a steambath (A1.1.2).

8.4.4.1 Add 20 mL of concentrated HNO₃ carefully. Add distilled water and remove from heat if the reaction becomes too violent.

8.4.4.2 When the reaction with nitric acid has subsided, carefully add concentrated HF dropwise until no reaction is observed upon addition. Stop and add distilled water if the reaction becomes too violent; then resume acid addition when the reaction subsides.

8.4.4.3 Allow the solution to evaporate to dryness on the steambath.

8.4.4.4 Repeat the steps given in 8.4.4.1 through 8.4.4.3 until no further reduction in the amount of solids is observed when the sample is taken to dryness.

8.4.4.5 Ignite in a muffle furnace at 700°C for 1 to 2 h.

8.4.4.6 Allow the dish to cool, cover with a watch glass, and then proceed to 8.4.5.

8.4.5 *Nitric Acid Sample Leach*—Add 100 to 150 mL of 8 M HNO₃ to the beaker or dish carefully. Add distilled water if the reaction becomes too violent.

8.4.5.1 Heat the beaker or dish on a steam bath while it is covered with a watch glass.

8.4.5.2 Allow the solution to cool; then filter (A1.1.3 – A1.1.6) into a beaker. Retain the filtrate until it is appropriate to combine it with the solution(s) resulting from dissolution of the residue.

8.4.5.3 Transfer the filter paper to a platinum crucible or dish.

8.4.5.4 Perform a sodium carbonate fusion using the step given in 8.5.2 or, if necessary, other treatments of the residue as identified in references.

8.5 *Dissolution of Refractory Uranium-Containing Material by Carbonate Fusion*—Common dissolution techniques are described in Annex A1. The techniques are referenced to the appropriate section by a superscript at the first place in the procedure where they may be applicable.

8.5.1 *Sample Preparation*—This procedure is used to dissolve refractory uranium-containing materials to provide solutions for analysis. The material is usually a residue from the

acid digestion described in 8.4. If this is not a residue, then first obtain the mass of the sample (usually using an analytical balance with 0.1-mg sensitivity). Transfer the uranium-containing material quantitatively to a platinum crucible or dish.

8.5.1.1 If the sample is in filter paper, dry and then ignite it in a muffle furnace at 700°C for 1 to 2 h or until no carbon is visible. (The sample may foam during fusion if carbon is not removed.) If the ignition does not remove carbon completely, digest the sample with several drops of concentrated H₂SO₄ and fume to dryness on a hot plate.

8.5.1.2 Allow the crucible to cool to ambient temperature (A1.1.8) and then proceed to 8.5.2.

8.5.2 *Sodium Carbonate Fusion*—Add a volume of Na₂CO₃ (1 to 10 g) to the platinum crucible or dish that is approximately 5 to 10 times the volume of residue.

8.5.2.1 Stir the mixture carefully with a glass or platinum stirrer, and place the stirrer in a beaker (A1.1.1) labeled with the crucible or dish number.

8.5.2.2 Cover the crucible or dish with a platinum lid, (A1.1.7) and place it on a triangle supported by a tripod in a hood. Heat the crucible or dish carefully with a flame from a blast burner until the flux melts; then increase the air supply to attain maximum temperature. If proper safety precautions are followed, the crucible may instead be heated in a muffle furnace set initially at 300°C and then increased to 900°C.

8.5.2.3 Using platinum-tipped tongs, remove the lid and swirl the crucible or dish contents carefully to mix its contents.

8.5.2.4 Replace the lid and continue heating for 3 to 5 h. Repeat swirling occasionally (8.5.2.3) during the 3 to 5 h heating period.

8.5.2.5 Remove the crucible or dish from the flame, swirl carefully until the flux solidifies, and allow it to cool to ambient temperature (A1.1.8).

8.5.2.6 If necessary, (A1.1.9) wipe off the exterior of the crucible or dish with a moistened towel. Place the crucible or dish and lid in the beaker from the step given in 8.5.2.1.

8.5.2.7 Cover the crucible or dish and lid with distilled water. Add concentrated HNO₃ slowly and carefully until the reaction stops, covering the beaker with a watch glass after each addition of acid. Remove and rinse the crucible or dish, lid, and stirring rod into the beaker with 8 M HNO₃. Set the crucible or dish aside for reuse.

8.5.2.8 Add 10 mL of concentrated HNO₃, and heat the covered beaker on the steam bath for 1 to 2 h.

8.5.2.9 If it is necessary to reduce the volume at this point, remove the watch glass cover and continue heating until the desired volume is obtained.

8.5.2.10 Remove the beaker from the steam bath, allow the solution to cool, and examine carefully for a residue.

8.5.2.11 If the sample has dissolved completely, combine the resulting solution with any other solutions from the dissolution of this material. Proceed as in 8.2.5.

8.5.2.12 If the solution is cloudy or contains solids, filter (A1.1.3 – A1.1.6) it into a beaker. This may be a beaker that contains a previous filtrate or a new beaker.

8.5.2.13 Place the filter paper in a platinum crucible or dish. Dry the filter paper, and ignite it in a muffle furnace at 700°C for 1 to 2 h, or until no carbon is visible.

8.5.2.14 Allow the crucible or dish to cool to ambient temperature; then add 10 mL of concentrated HNO₃ and 1 mL of concentrated HF to the dish, and heat to dryness on the steambath in order to remove any silica.

8.5.2.15 Repeat the step given in 8.5.2.14 until no further reduction in the amount of residue present is observed.

8.5.2.16 Add 5 to 10 mL of 8 M HNO₃, and heat on a steam bath to dissolve the residue.

8.5.2.17 If the solution is clear, add it to the filtrate. Proceed as in 8.2.5.

8.5.2.18 If the solution is not clear, evaporate it to dryness and repeat the steps given in 8.5.2.1 – 8.5.2.17 once, then proceed as in 8.2.4.11 – 8.2.4.13.

8.6 *Dissolution of Uranium Materials Using a Microwave Oven and High Pressure, Heavy Duty Dissolution Vessels (HDV)*:

8.6.1 Pre-clean digestion vessels by soaking in 8 MHNO₃ and rinsing with Type 1 Reagent Grade water.

8.6.2 Quantitatively transfer a sample containing approximately 1.0 g of uranium to a clean polytetrafluoroethylene liner of a HDV. Scrap or impure samples may require a smaller sample volume.

8.6.3 Add 8 mL of 8MHNO₃, 3 mL of 12M HCl, and 2 mL of 7.2M HF acids to the sample in the HDV.

NOTE 7—The rupture membranes must be replaced with new ones. Failure to do so may cause premature rupturing and loss of sample. Do not double the membranes for safety reasons.

8.6.4 Clean the fittings and replace the rupture membrane in the lid of the HDV with a new one.

8.6.5 Assemble the HDV maintaining the identity of the sample in the HDV. Place the heaviest or most reactive matrix material in the pressure sensing position for system control. The acids used in this procedure may attack the pressure transducer and become contaminated; do not use the pressure sensing position for a sample aliquant to be reported for impurities. Hand tighten the lids and then retighten with a spanner wrench.

NOTE 8—After assembly, check for clearance between cap and body (1.6 mm (1/16 in.) to 3.2 mm (1/8 in.) is normal). If no gap exists, disassemble, add another heat shield spacer to the assembly, and reassemble.

8.6.6 Repeat steps 8.6.1 – 8.6.5 for each sample.

8.6.7 Prepare the microwave oven according to manufacturer instructions.

8.6.8 Place the remaining HDVs on the turntable in a balanced fashion. If an odd number of samples is to be digested, use an empty HDV (cap not tightened) for balance.

8.6.8.1 **Warning**—Rotate the turntable through several 360° rotations of the carousel. Check to make sure the lines are not tangled and to preserve the integrity of safety features.

8.6.9 The digestion parameters will vary depending upon the composition of the sample. Load the appropriate dissolution program and then run. This program must be determined

by the laboratory procedure using guidance from the microwave system manufacturer.

8.6.9.1 Warning—If the rupture disc fails in the pressure sensing vessel (PSV), the microwave will automatically stop, but if it fails in one of the other HDVs the microwave may or may not stop. Press the stop button and wait at least one hour before removing the vessels. Do not open the microwave for at least fifteen minutes after stopping because another disc may fail, spewing hot acid.

8.6.10 For pure UO₂ samples, wait until the pressure reaches ambient pressure (about 1 h) before opening the microwave door. For impure scrap samples, use temperature and stable pressure to determine when vessels have stabilized, the bleed open carefully in a hood or the ventilated microwave enclosure to remove the pressure.

8.6.10.1 Warning—If any pressure remains in the vessels when they are opened, then hot acidic fumes or liquids will spew forth endangering the operator.

8.6.11 Remove the non pressure sensing vessels and place them in the fume hood.

8.6.12 Remove the fitting from the PSV and move it to the fume hood.

NOTE 9—If volatile elements are to be determined in subsequent analysis, then samples should be cooled to room temperature before opening the vessels.

8.6.13 Quantitatively transfer the sample to a clean, 50 mL volumetric flask; rinse the lid and polytetrafluoroethylene liner several times, and dilute to volume.

8.6.14 Cap the volumetric flask and mix thoroughly.

8.6.15 Solutions may be transferred after mixing to pre-leached 60 mL plastic bottles or equivalent container.

8.6.16 Repeat steps **8.6.13–8.6.15** for each vessel.

9. Reliability

9.1 The objectives of a dissolution treatment are complete solubility, absence of residues, and complete recovery in all operations. Careful laboratory practices are required in all operations.

9.2 For optimum reliability, every dissolved sample solution must be inspected carefully for solids deposited on the container bottom and in suspension. The observation of suspended particles is enhanced by shining a light beam into the solution from the side and looking down on the solution. Alternatively, the solution can be centrifuged to concentrate the residue.

10. Precision and Bias

10.1 This is not a test method, and no data are generated by this practice, so a precision and bias statement is not required.

11. Keywords

11.1 dissolution; microwave dissolution; microwave oven; uranium-aluminum alloy; uranium ash; uranium dissolution; uranium metal; uranium oxide; uranium scrap

ANNEX

(Mandatory Information)

A1. COMMON TECHNIQUES

A1.1 Techniques that may be useful during the dissolution of samples and helpful suggestions are indicated below:

A1.1.1 Label beakers with a pencil on the matte-finish enameled area. The label may be erased easily but will not be lost during processing of the sample.

A1.1.2 Samples are normally heated on a steambath during acid dissolution. Steambath heating has the advantage that samples will not be lost due to “bumping,” even if they are not watched closely. Samples may be heated on a hot plate set at low temperature, provided that they are watched continuously to prevent boiling, bumping, or evaporation to dryness.

A1.1.3 To avoid clogging filter paper when filtering samples that contain very fine particulate, add filter paper pulp to the filter paper in the funnel.

A1.1.4 To eliminate splashing during the filtration of samples, position the funnel so that the tip of the stem touches the side of the beaker.

A1.1.5 All transfers and all filtrations during dissolution procedures must be quantitative. This normally includes three to four rinses with distilled water or dilute acid of all labware, including stirring rods, funnels, watch glasses, beakers, crucible lids, crucibles, etc. To facilitate the transfer of residues from beakers to filter papers during filtration, wipe the inside of the beaker by pushing a small piece of filter paper around the walls and bottom with a rubber policeman on a glass stirring rod. Add the filter paper to the funnel, and rinse the beaker and policeman with distilled water into the funnel.

A1.1.6 Glass stirring rods may be used to help perform transfers from one container to another without loss of the sample by splashing. The solution is allowed to flow down the glass stirring rod during the transfer.

A1.1.7 When ignitions or fusions must be performed, record the crucible or dish number not only in the notebook, but also on the corresponding sample beaker. This will help to prevent improper transfers, which result in the loss of samples.



A1.1.8 The platinum crucible or dish may be placed on a heat dissipating board while it is cooling.

A1.1.9 To prevent contamination of the sample, the exterior of the crucible or dish must either be kept clean or be cleaned prior to immersing it in acid.

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