



Designation: C1347 – 08 (Reapproved 2014)^{ε1}

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.

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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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.

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 (29*M*), 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.