Designation: C 1347 – 02

Standard Practice for Preparation and Dissolution of Uranium Materials for Analysis[1]

This standard is issued under the fixed designation C 1347; 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 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 the standard. The values given in parentheses are for information only.

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:*

C 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder[2]

C 776 Specification for Sintered Uranium Dioxide Pellets[2]

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis[2]

D 1193 Specification for Reagent Water[3]

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 C 1168. 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.[4,5,6] 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.

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.

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;

[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] *Annual Book of ASTM Standards*, Vol 12.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

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

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

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

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 C 753 and C 776. 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 oven[7] 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.

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 µL*—5 mL (or equivalent). Accuracy of ± 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), 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 such specifications are available.[8] 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 Grade[9] water may be required dependent upon the accuracy and precision of the analysis method used.

6.3 *Nitric Acid* (HNO_3), concentrated (sp gr 1.4), 16 *M* .

6.4 *HNO_3, 8 M*—Add 500 mL of concentrated HNO_3 (sp gr 1.4) to approximately 400 mL of water and dilute to 1 L.

6.5 *HNO_3, 10 %* Add 100 mL of concentrated HNO_3(sp gr 1.4) to 800 mL. Type 1 Reagent Grade water and dilute to 1 L.

6.6 *HNO_3, 2 %* Add 20 mL of concentrated HNO_3 to 900 mL. Type 1 Reagent Grade water and dilute to 1 L.

6.7 *Hydrochloric Acid* (HCI), 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* (H_2SO_4), concentrated 18 *M* (sp gr 1.8).

6.11 *Sulfuric Acid*, 9 *M*—Add 500 mL of concentrated (sp gr 1.8) H_2SO_4 to approximately 400 mL of water, cool and dilute to 1 L. Store in a glass bottle.

6.12 *Sodium Carbonate* (Na_2CO_3).

6.13 *Sodium Bisulfate* ($NaHSO_4$).

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

[7] 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 which you may attend.

[8] *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.

[9] See Specification D 1193.