



Designation: C1347 – 08 (Reapproved 2023)

Standard Practice for Preparation and Dissolution of Uranium Materials for Analysis¹

This standard is issued under the fixed designation C1347; 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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 7.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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.

Current edition approved Jan. 1, 2023. Published January 2023. Originally approved in 1996. Last previous edition approved in 2014 as C1347 – 08 (2014) ^{ϵ 1}. DOI: 10.1520/C1347-08R23.

2. Referenced Documents

2.1 ASTM Standards:²

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C776 Specification for Sintered Uranium Dioxide Pellets for Light Water Reactors

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.

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

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.

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.

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.

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 µL—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 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.

⁷ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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.