



Designation: C1168 – 23

Standard Practice for Preparation and Dissolution of Plutonium Materials for Analysis¹

This standard is issued under the fixed designation C1168; 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 is a compilation of dissolution techniques for plutonium materials that are applicable to the test methods used for characterizing these materials. Dissolution treatments for the major plutonium materials assayed for plutonium or analyzed for other components are listed. Aliquots of the dissolved materials are dispensed on a mass basis when one of

the analyses must be of high precision, such as plutonium assay; otherwise they are dispensed on a volume basis.

1.2 Procedures in this practice are intended for the dissolution of plutonium metal, plutonium oxide, and uranium-plutonium mixed oxides. Aliquots of dissolved materials are analyzed using test methods, such as those developed by Subcommittee C26.05 on Methods of Test, to demonstrate compliance with applicable requirements. These may include product specifications such as Specifications C757 and C833.

1.3 One or more of the procedures in this practice may be applicable to unique plutonium materials, such as alloys, compounds, and scrap materials. The user must determine the applicability of this practice to such materials.

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

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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1.5 The values stated in SI units are to be regarded as standard. The non-SI unit of molarity (M) is also to be regarded as standard. Values in parentheses (non-SI units), where provided, are for information only and are not considered standard.

1.6 *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.*

1.7 *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.*

2. Referenced Documents

2.1 ASTM Standards:²

C757 Specification for Nuclear-Grade Plutonium Dioxide Powder for Light Water Reactors

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets for Light Water Reactors

C859 Terminology Relating to Nuclear Materials

C1145 Terminology of Advanced Ceramics

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within Nuclear Industry

D1193 Specification for Reagent Water

D7439 Test Method for Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma–Mass Spectrometry

E1154 Specification for Piston or Plunger Operated Volumetric Apparatus and Operator Qualification

2.2 ISO Standards:³

ISO 8655 Piston-Operated Volumetric Instruments (6 parts)

3. Terminology

3.1 *Definitions*—Except as otherwise defined herein, definitions of terms are as given in Terminology **C859**.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calcination, n*—(**calcine**, *v*)—firing or heating of a granular or particulate solid at less than fusion temperature, but sufficient to remove most of its chemically combined volatile matter and otherwise to develop the desired properties for use.

C1145

3.2.2 *reagent blank, n*—solution containing all reagents used in sample preparation, in the same quantities used for preparation of blank and sample solutions.

D7439

4. Summary of Dissolution Procedures

4.1 Most plutonium-containing materials are dissolved with various mineral acids and, except for plutonium metal, with applied heat. In some cases, dissolution-resistant materials are dissolved in heated and sealed containers in which pressurization provides a higher temperature than is attained at ambient pressure.

4.2 The dissolved materials are quantitatively transferred to a tared polyethylene dispensing bottle for subsequent deliveries of weighed aliquots for high-precision analysis methods, such as assays, or to a volumetric flask for deliveries of volume aliquots for less precise analysis methods, such as impurity analyses. Dilute acids are used as rinses to effect quantitative transfers and as diluents in the polyethylene dispensing bottles and volumetric flasks. The use of water for these purposes can, in some cases, result in hydrolysis of plutonium to produce polymers that, although soluble, are nonreactive in separation treatments or in plutonium assay methods that have no pretreatments, such as fuming with acid.

4.3 The procedures included in this practice are briefly described in 4.4 through 4.8. Other dissolution reagents are possible, but are not addressed in this practice. When alternate reagents are employed, the user shall verify their suitability for the intended use.

4.4 Plutonium metal is dissolved with hydrochloric acid (Section 9 or 10) or with sulfuric acid (Section 11).

4.5 Plutonium oxide, calcined at about 1000 °C or lower, is dissolved with a mixture of hydrochloric, nitric, and hydrofluoric acids using the sealed-reflux technique (Section 12) (1).⁴ More refractory plutonium oxide is dissolved with a fusion using sodium bisulfate flux (Section 13) (2).

4.6 A mixture of nitric and hydrofluoric acid in beakers can be used for low-fired (<650 °C) plutonium oxide (Section 14). Open-vessel dissolution using nitric and hydrofluoric acids can be used for low-fired plutonium oxide (Section 15) or mixed oxide (Section 16). Closed-vessel hot block dissolution can also be used for low-fired plutonium oxide (Section 17). Plutonium oxide fired at temperatures above 650 °C may also be dissolved using one of the methods described in Sections 14 through 17 when complete dissolution can be demonstrated.

4.7 Uranium-plutonium mixed oxide is dissolved in any of the following ways: by the sealed-reflux technique using a mixture of hydrochloric, nitric, and hydrofluoric acids (Section 12), by sodium bisulfate fusion (Section 13), by a heated mixture of nitric and hydrofluoric acids in a beaker (Section 14), or by open-vessel dissolution using nitric and hydrofluoric acids (Section 16).

4.8 Mixed oxide pellets are dissolved using open-vessel dissolution with nitric and hydrofluoric acids (Section 18).

4.9 Combinations of these dissolution techniques described in the preceding paragraphs are sometimes used on difficult-to-dissolve samples.

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 Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

⁴ The boldface numbers in parentheses refer to a list of references at the end of this practice.