Designation: C1168 – 15

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

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 samples are dispensed on a weight basis when one of the analyses must be highly reliable, 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 samples 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:

Procedure Number	Procedure Title	Section
1	Dissolution of Plutonium Metal with Hydrochloric Acid at Room Temperature	9
2	Dissolution of Plutonium Metal with Hydrochloric Acid and Heating	10
3	Dissolution of Plutonium Metal with Sulfuric Acid	11
4	Dissolution of Plutonium Oxide and Uranium-Plutonium Mixed Oxide by the Sealed-Reflux Technique	12
5	Dissolution of Plutonium Oxide and Uranium-Plutonium Mixed Oxides by Sodium Bisulfate Fusion	13
6	Dissolution of Uranium-Plutonium Mixed Oxides and Low-Fired Plutonium Oxide in Beakers	14
7	Open-Vessel (with Reflux Condenser) Dissolution of Plutonium Oxide Powder	15
8	Open-Vessel (with Reflux Condenser) Dissolution of Mixed Oxide Powder	16
9	Closed-Vessel Hot Block Dissolution of Plutonium Oxide Powder	17
10	Open-Vessel (with Reflux Condenser) Dissolution of Mixed Oxide Pellets	18

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[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. 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets

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 the 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

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 samples 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 techniques (Section 12) **(1).**[4] More refractory plutonium oxide is dissolved with a fusion using sodium bisuflate 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.

4.10 Quantitative transfers of samples and subsequent solution are required. Whenever a loss is incurred or even suspected, the sample is rejected. Solutions of dissolved samples are inspected for undissolved particles; if particles are present, further treatment is necessary to attain complete solubility. When analyzing the dissolved sample for trace impurities, caution should be exercised so the dissolution

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[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.

process does not cause the impurity to be lost or does not significantly increase the level of impurity being determined.

5. Significance and Use

5.1 Plutonium and uranium mixtures are used as nuclear reactor fuels. For use as a nuclear reactor fuel, the material must meet certain criteria for combined uranium and plutonium content, effective fissile content, and impurity content as described in Specifications C757 and C833. After dissolution using one of the procedures described in this practice, the material is assayed for plutonium and uranium to determine if the content is correct as specified by the purchaser.

5.2 Unique plutonium materials, such as alloys, compounds, and scrap metals, are typically dissolved with various acid mixtures or by fusion with various fluxes. Many plutonium salts are soluble in hydrochloric acid. One or more of the procedures included in this practice may be effective for some of these materials; however their applicability to a particular plutonium material shall be verified by the user.

6. Apparatus

6.1 Ordinary laboratory apparatus are not listed, but are assumed to be present.

6.2 The following items are used by most or all procedures listed herein. Apparatus specific to one, or a few, procedures are listed under the specific procedures where they are used.

6.2.1 *Balances,* for weighing samples and solutions. A balance with a sensitivity of 0.1 mg is necessary; however, a balance with 0.01 mg sensitivity is more desirable. A calibrated balance must be used.

6.2.2 *Beakers and Erlenmeyer Flasks*—Generally, borosilicate glass is recommended; however, the analyst should be sure that safety and sample contamination from the container are considered when choosing appropriate containers. For example, when hydrofluoric acid is used, the use of borosilicate glass may be avoided due to etching.

6.2.3 *Volumetric flasks or polyethylene dispensing bottles,* for collecting the final solution from which aliquots are dispensed.

6.2.4 *File or brush,* for cleaning plutonium metal surfaces (used in Sections 9 through 11).

6.2.5 *Inert Atmosphere Glove-Box System*—capability of maintaining less than 10 ppm of H_2O and 2 % of O_2 is preferred.

6.2.6 *Piston-Operated Volumetric Pipettors,* complying with the requirements of Specification E1154, applicable portions of ISO 8655 (six parts), or both.

7. Reagents

NOTE 1—Reagents used in specific procedures are listed within the procedure or procedures where they are used. The following information applies to all reagents.

7.1 *Purity of Reagents*—All reagents used should be of the highest purity available. Other grades may be used if they are determined not to affect the final result.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, as defined by Type I of Specification D1193.

8. Precautions

8.1 Strong acids are used by these dissolution procedures. Safety glasses and gloves must be worn when handling these solutions. Extreme care should be exercised in using hydrofluoric acid and other hot concentrated acids. Acid solutions are evaporated by these dissolution procedures. These operations must be conducted in a fume hood.

8.2 Hydrofluoric acid is a highly corrosive and toxic acid that can severely burn skin, eyes, and mucous membranes. 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. Familiarization and compliance with the Safety Data Sheet is essential.

8.3 Extreme care is required when using procedures that involve closed vessels, pressurization, or both (Sections 12 and 17). Potential explosions or rupturing of vessels could injure personnel or breach glovebox containment, or both. Appropriate controls are required to prevent pressure or temperature from exceeding prescribed by safety limits.

9. Procedure 1 – Dissolution of Plutonium Metal with Hydrochloric Acid at Room Temperature

9.1 *Apparatus:*

9.1.1 *Hot Plate,* thermostatically controlled, capable of maintaining a surface temperature of up to at least 200°C.

NOTE 2—The surface temperature of a hot plate can vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized before use.

9.1.2 *Centrifuge Tube,* optional, to receive weighed sample if beaker is not used.

9.1.3 *Watch glasses.*

9.1.4 *Infrared lamp.*

9.2 *Reagents:*

9.2.1 *Hydrochloric Acid,* concentrated (sp gr 1.18), 12 *M*.

9.2.2 *Hydrochloric Acid,* 6 *M*—Add 500 mL of 12 *M* HCl to <500 mL of water and dilute to 1 L with water.

9.2.3 *Hydrochloric Acid,* 1 *M*—Add 83 mL of 12 *M* HCl to <900 mL of water and dilute to 1 L with water.

9.2.4 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

9.2.5 *Hydrofluoric Acid,* 1.3 *M*—Transfer 4.8 mL of 28 *M* HF, using a plastic pipet, to a 100-mL plastic graduated cylinder containing <90 mL of water and dilute to 100 mL with water. Transfer to a plastic bottle for storage.

9.2.6 *Nitric Acid,* concentrated (sp gr 1.42), 16 *M*.

9.3 *Procedure:*

9.3.1 Remove surface oxide, if present, from the bulk sample before cutting into portions. Removal of surface oxide may be performed by filing or brushing the plutonium metal, or by using other mechanical or chemical means.

NOTE 3—Plutonium metal reacts with air and moisture to form PuO_2. An inert-atmosphere glovebox system can be utilized for mechanical removal of surface oxide, but is not recommended for removal by chemical means. Use of an inert-atmosphere glovebox system is recommended when preparing standards or tracers.

9.3.2 Weigh a representative sample size, considering the required uncertainty and the analysis method to be used. Normal sample size is 50 to 700 mg, but this may vary. A certified sample mass, if known, may be used in lieu of weighing.

9.3.3 Transfer the weighed sample to a beaker or centrifuge tube and cover with a watch glass.

9.3.4 Add 6 *M* HCl dropwise, or in small portions, through the spout of the beaker or centrifuge tube until the sample dissolves completely.

9.3.5 Inspect the solution for suspended particles or deposited solid and, if present, warm the solution (using, for example, an infrared lamp).

9.3.6 If solid still is present, add 0.5 mL 16 *M* HNO_3 and three drops 1.3 *M* HF, and heat until dissolution is complete.

9.3.7 Quantitatively transfer the solution using at least four rinses of the watch glass and beaker or centrifuge tube with 1 *M* HCl. Transfer to a volumetric flask and bring to volume using 1 *M* HCl. Alternatively, for high-precision analyses, transfer to a previously tared polyethylene dispensing bottle, weigh the bottle with the solution, and calculate the weight difference to determine final volume.

9.3.8 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

10. Procedure 2 – Dissolution of Plutonium Metal with Hydrochloric Acid and Heating

10.1 This procedure is for Pu metal pieces of approximately 0.5 g mass which may be conditioned under an inert atmosphere glovebox system.

10.2 *Apparatus:*

10.2.1 *Hot Plate:* thermostatically controlled, capable of maintaining a surface temperature of up to at least 200°C.

NOTE 4—The surface temperature of a hot plate can vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized before use.

10.2.2 *Watch glasses.*

10.3 *Reagents:*

10.3.1 *Hydrochloric Acid,* concentrated (sp gr 1.18), 12 *M*.

10.3.2 *Hydrochloric Acid,* 1 *M*—Add 83 mL of 12 *M* HCl to <900 mL of water and dilute to 1 L with water.

10.3.3 *Hydrochloric Acid,* 0.1 *M*—Add 8.3 mL of 12 *M* HCl to <900 mL of water and dilute to 1 L with water.

10.3.4 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

10.3.5 *Hydrofluoric Acid,* 1 *M*—Transfer 3.6 mL of 28 *M* HF, using a plastic pipet, to a 100-mL plastic graduated cylinder containing <90 mL of water and dilute to 100 mL with water. Transfer to a plastic bottle for storage.

10.4 *Procedure:*

10.4.1 Remove surface oxide, if present, from the bulk sample before cutting into portions. Removal of surface oxide may be performed by filing or brushing the plutonium metal, or by using other mechanical or chemical means.

NOTE 5—Plutonium metal reacts with air and moisture to form PuO_2. An inert-atmosphere glovebox system can be utilized for mechanical removal of surface oxide, but is not recommended for removal by chemical means. Use of an inert-atmosphere glovebox system is recommended when preparing standards or tracers.

10.4.2 Weigh a representative sample size, considering the required uncertainty and the analysis method to be used. A certified sample mass, if known, may be used in lieu of weighing.

10.4.3 Transfer the weighed sample to a beaker or Erlenmeyer flask, add just enough 0.1 *M* HCl to cover the metal, then slowly add 7 to 8 mL of 12 *M* HCl and cover with a watch glass.

10.4.4 Allow the metal to dissolve for 10 to 15 min at room temperature.

10.4.5 Inspect the solution for suspended particles or deposited solid and, if present, add an additional 2 mL of 12 *M* HCl and heat to boiling for 2 h.

10.4.6 If solid is still present, add an additional 2 mL of 12 *M* HCl and 2 drops of 1*M* HF, and heat for an additional 2 h.

10.4.7 Repeat step 10.4.6 as necessary until dissolution is complete.

10.4.8 Quantitatively transfer the solution using at least four rinses of the watch glass and beaker or centrifuge tube with 1 *M* HCl. Transfer to a volumetric flask and bring to volume using 1 *M* HCl. Alternatively, for high-precision analyses, transfer to a previously tared polyethylene dispensing bottle, weigh the bottle with the solution, and calculate the weight difference to determine final volume.

10.4.9 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

11. Procedure 3 – Dissolution of Plutonium Metal with Sulfuric Acid

11.1 *Apparatus:*

11.1.1 *Infrared lamp.*

11.1.2 *Watch glasses.*

11.1.3 *Magnetic stirrer* with TFE-fluorocarbon stir bar.

11.2 *Reagents:*

11.2.1 *Sulfuric Acid,* concentrated (sp gr 1.84), 18 *M*.

11.2.2 *Sulfuric Acid,* 0.5 *M*—Cautiously add 28 mL of 18 *M* H_2SO_4 to water and dilute to 1 L with water.

11.3 *Procedure:*

11.3.1 Remove surface oxide, if present, from the bulk sample before cutting into portions. Removal of surface oxide may be performed by filing or brushing the plutonium metal, or by using other mechanical or chemical means.

NOTE 6—Plutonium metal reacts with air and moisture to form PuO_2. An inert-atmosphere glovebox system can be utilized for mechanical removal of surface oxide, but is not recommended for removal by chemical means. Use of an inert-atmosphere glovebox system is recommended when preparing standards or tracers.

11.3.2 Weigh a representative sample size, considering the required uncertainty and the analysis method to be used. Normal sample size is 50 to 700 mg, but this may vary. A certified sample mass, if known, may be used in lieu of weighing.

11.3.3 Transfer the weighed sample to a beaker or Erlenmeyer flask and cover with a watch glass.

11.3.4 Carefully place a TFE-fluorocarbon stirring bar in the beaker or flask along with 30 to 40 mL of 0.5 *M* H_2SO_4, put the dissolution container on a magnetic stirrer, and mix the solution until the sample is dissolved.

11.3.5 Inspect the solution for suspended particles or deposited solid and, if present, warm the solution (using, for example, an infrared lamp).

11.3.6 Quantitatively transfer the solution using at least four rinses of the watch glass and beaker or Erlenmeyer flask with 0.5 *M* H_2SO_4. Transfer to a volumetric flask and bring to volume using 0.5 *M* H_2SO_4. Alternatively, for high-precision analyses, transfer to a previously tared polyethylene dispensing bottle, weigh the bottle with the solution, and calculate the weight difference to determine final volume.

11.3.7 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

12. Procedure 4 – Dissolution of Plutonium Oxide and Uranium-Plutonium Mixed Oxide by the Sealed-Reflux Technique

NOTE 7—The procedure described in this section is based on Ref. (**1**) and is intended for sample sizes of approximately 0.5 g. A modification of the procedure and apparatus for samples of up to 100 g is described in Ref. (**3**). The procedure has been used on mixed oxide pellets with minor modifications; see Note 10.

12.1 *Apparatus:*

12.1.1 *Sealed-Reflux Dissolution Apparatus*—The example apparatus is shown in Fig. 1 and Fig. 2 and is further described in Ref (**1**).

NOTE 8—The apparatus shown in Fig. 1 and Fig. 2 is based on Ref. (**1**) and should be considered an example. A different apparatus may be used if it has been shown to meet the performance requirements of this section.

12.1.2 *Stopper*—A stopper which will not react with the dissolution matrix must be used. A solid stopper inserted in a hollow, polyethylene stopper liner has been found to be satisfactory. Certain fluorinated elastomers, designated as FKMs, have also been found to be satisfactory.

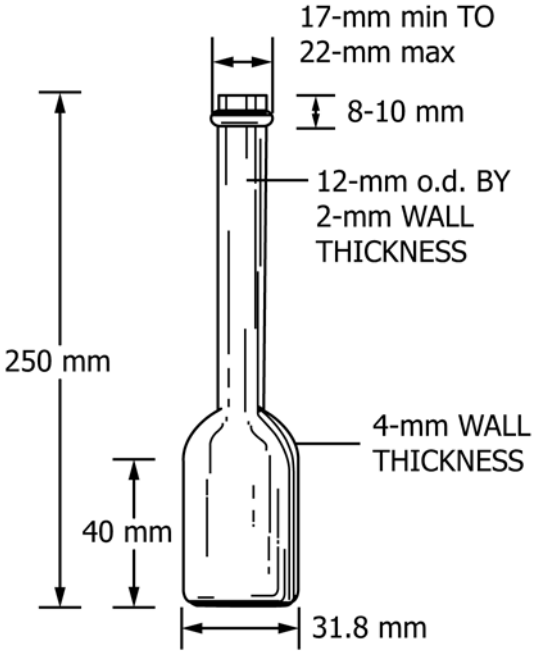

FIG. 1 Sealed-Reflux Dissolution Tube

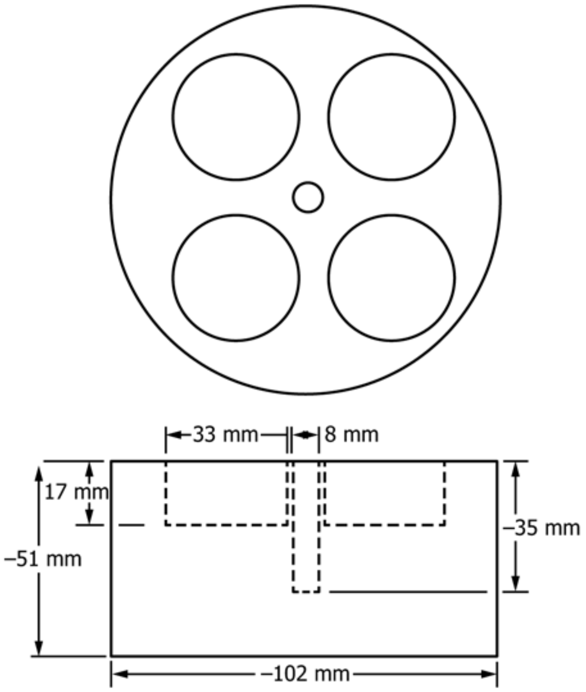

FIG. 2 Heating Block

12.1.3 *Spring clamp* that will hold the tube snugly to the reflux condenser, but will release pressure at a level well below the failure point of the tube. (**Warning—**The spring clamp must be selected to ensure that the tube releases pressure well below the point that would cause the tube to fail physically. A suggested value is 827 kPa (120 psi) but should be verified prior to use. Only one clamp should be used so as not to compromise this safety feature.)

12.1.4 *Weighing pan.*

12.2 *Reagents:*

12.2.1 *Hydrochloric Acid,* concentrated (sp gr 1.18), 12 *M*.

12.2.2 *Hydrochloric Acid,* 1 *M*—Add 83 mL of 12 *M* HCl to <900 mL of water and dilute to 1 L with water.

12.2.3 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

12.2.4 *Hydrofluoric Acid,* 1.3 *M*—Transfer 4.8 mL of 28 *M* HF, using a plastic pipet, to a 100-mL plastic graduated cylinder containing <90 mL of water and dilute to 100 mL with water. Transfer to a plastic bottle for storage.

12.2.5 *Nitric Acid,* concentrated (sp gr 1.42), 16 *M*.

12.3 *Procedure:*

12.3.1 Tare a weighing pan or other type of container.

12.3.2 Transfer sample to the tared pan or other container until the desired weight of sample is obtained, usually 0.5 g. Weigh to at least 0.1 mg sensitivity.

12.3.3 Quantitatively transfer the sample from the pan into a sealed-reflux tube, see Fig. 1.

12.3.4 Reweigh the pan. Compute the weight of sample transferred to the tube by subtracting the weight of the pan from the weight of the sample plus the pan.

12.3.5 Add 5 mL of 12 *M* HCl, 3 drops 16 *M* HNO_3, and 3 drops 1.3 *M* HF to the tube.

NOTE 9—Other acid combinations containing nitric or sulfuric acid as the major constituent that can be used are described in Ref. (**1**).

12.3.6 Seal the tube with a stopper, spring clamp, and heat at 150°C in a heating block (see Fig. 2) for 2 h or until the sample dissolves completely. Cool the tube to <30°C before releasing the clamp. (**Warning—**Heating at temperatures above 150°C can result in overpressurization of the tube.)

NOTE 10—This procedure has been used to dissolve mixed (uranium-plutonium) oxide pellets with a heating time of 12 to 16 h and five drops of 1.3 *M* HF. (**4**)

12.3.7 Quantitatively transfer the solution using at least four rinses of the watch glass and beaker or Erlenmeyer flask with 1 *M* HCl. Transfer to a volumetric flask and bring to volume using 1 *M* HCl. Alternatively, for high-precision analyses, transfer to a previously tared polyethylene dispensing bottle, weigh the bottle with the solution, and calculate the weight difference to determine final volume.

12.3.8 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

13. Procedure 5 – Dissolution of Plutonium Oxide and Uranium-Plutonium Mixed Oxide by Sodium Bisulfate Fusion

13.1 This dissolution procedure is not recommended for use with trace-impurity analysis methods because of the possibility of contaminating the sample with impurities from the sodium bisulfate flux or beaker, or both.

13.2 *Apparatus:*

13.2.1 *Furnace,* capable of maintaining an even temperature of ±10°C up to 700°C.

NOTE 11—Use of a furnace with an automated timer is recommended.

13.2.2 *Watch glasses.*

13.3 *Reagents:*

13.3.1 *Sodium Bisulfate, Anhydrous, Fused,* $NaHSO_4$—Grind the sodium bisulfate to a fine powder just before use, if necessary.

13.3.2 *Sulfuric Acid,* concentrated (sp gr 1.84), 18 *M*.

13.3.3 *Sulfuric Acid,* 0.5 *M*—Cautiously add 28 mL of 18 *M* H_2SO_4 to water and dilute to 1 L with water.

13.4 *Procedure:*

13.4.1 Quantitatively transfer a 100 to 300-mg sample of plutonium oxide or 1 to 1.5 g of pulverized uranium-plutonium mixed oxide to a beaker or Erlenmeyer flask. A fused silica container is recommended to reduce sample contamination. The sample should be weighed to at least ±0.1 mg.

NOTE 12—Because of the recommended temperature, fused silica or 96 % silica are the container materials of choice; however, experience has shown that borosilicate glass can be used.

13.4.2 Add ten times the sample weight of anhydrous fused $NaHSO_4$ and carefully swirl the beaker to mix the powders.

13.4.3 Cover the beaker with a watch glass and place it in a furnace at room temperature.

13.4.4 Slowly heat to 600 to 625°C to prevent sample spatter or temperature overshoot, or both, hold at 600 to 625°C for 20 to 30 min. Do not exceed 600 to 625°C or the 30 min time limit because an insoluble component may form.

13.4.5 After cooling at room temperature for at least 30 min, add 30 to 40 mL of 0.5 *M* H_2SO_4 or of water.

NOTE 13—Experience has shown that in this acid bisulfate medium, water can be used to dissolve the salt when maximum acid strength and sample volume cannot be exceeded. The solution should be mixed immediately after the water is added.

13.4.6 Quantitatively transfer the solution using at least four rinses of the watch glass and beaker or Erlenmeyer flask with 0.5 *M* H_2SO_4. Transfer to a volumetric flask and bring to volume using 0.5 *M* H_2SO_4. Alternatively, for high-precision analyses, transfer to a previously tared polyethylene dispensing bottle, weigh the bottle with the solution, and calculate the weight difference to determine final volume.

13.4.7 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

14. Procedure 6 – Dissolution of Uranium-Plutonium Mixed Oxides and Low-Fired (<650°C) Plutonium Oxide in Beakers

14.1 *Apparatus:*

14.1.1 *Hot Plate,* thermostatically controlled, capable of maintaining a surface temperature of up to at least 200°C.

NOTE 14—The surface temperature of a hot plate can vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized before use.

14.1.2 *Watch glasses.*

14.2 *Reagents:*

14.2.1 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

14.2.2 *Nitric Acid,* concentrated (sp gr 1.42), 16 *M*.

14.2.3 *Hydrofluoric Acid-Nitric Acid Mixture,* 0.05 *M* HF-16 *M* HNO_3—Add 1.8 mL 28 *M* HF, using a plastic pipet, to 1 L of 16 M HNO_3.

14.2.4 *Nitric Acid,* 1 *M*—Add 63 mL of 16 *M* HNO_3 to <900 mL of water and dilute to 1 L with water.

14.3 *Procedure:*

14.3.1 Quantitatively transfer a sample of up to 1 g that is weighed to at least ±0.1 mg into a beaker.

14.3.2 Add 10 to 50 mL of 0.05 *M* HF–16 *M* HNO_3 mixture, cover with a watch glass, and heat using a hot plate until the sample dissolves completely.

NOTE 15—Experience has shown that a HNO_3 concentration in this acid mixture of 8 *M* or greater is adequate.

NOTE 16—If extended heating times are required, it may be necessary to add a few drops of HF to maintain the HF concentration level.

14.3.3 Quantitatively transfer the solution using at least four rinses of the watch glass and beaker or Erlenmeyer flask with 1 *M* HNO_3. Transfer to a volumetric flask and bring to volume using 1 *M* HNO_3. Alternatively, for high-precision analyses, transfer to a previously tared polyethylene dispensing bottle, weigh the bottle with the solution, and calculate the weight difference to determine final volume.

14.3.4 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

15. Procedure 7 – Open-Vessel Dissolution (with Reflux Condenser) of Plutonium Oxide Powder

15.1 *Apparatus:*

15.1.1 *Hot Plate,* thermostatically controlled, capable of maintaining a surface temperature of up to at least 200°C.

NOTE 17—The surface temperature of a hot plate can vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized before use.

15.1.2 *Perfluoroalkoxy alkane (PFA) polymer narrow-mouth hot plate bottle,* 125 mL.

15.1.3 *Reflux condenser.*

15.2 *Reagents:*

15.2.1 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

15.2.2 *Nitric Acid,* concentrated (sp gr 1.42), 16 *M*.

15.2.3 *Hydrofluoric Acid-Nitric Acid Mixture,* 0.07 *M* HF-14 *M* HNO_3—Add 2.5 mL of 28 *M* HF to 892 mL of 16 *M* HNO_3 and dilute to 1 L with water, adding water slowly to avoid splattering.

15.2.4 *Nitric Acid, 0.2 M*—Add 12.6 mL of 16 *M* HNO_3 to <900 mL of water and dilute to 1 L with water.

15.3 *Procedure:*

15.3.1 Weigh an empty 125 mL PFA polymer narrow-mouth hot plate bottle to the nearest 0.1 mg. Record the weight, m_1.

15.3.2 Transfer sample into the bottle until the desired weight of sample is obtained, usually 0.5 g. Weigh the bottle containing the PuO_2 to the nearest 0.1 mg and record the weight, m_2. Calculate the weight of sample transferred to the bottle, m_3, by subtracting the tare weight m_1 from m_2.

15.3.3 For each gram of sample, add 22 mL of 0.07 *M* HF—14 *M* HNO_3 mixture.

NOTE 18—For the usual sample size of 0.5 g, the volume of 0.07 *M* HF—14 *M* HNO_3 mixture will be 11 mL.

15.3.4 Weigh the hot plate bottle containing the PuO_2 and acid mixture to the nearest 0.1 mg. Record the weight, m_4.

15.3.5 Set a reflux condenser on the bottle and heat the solution to boiling on a hot plate until dissolution is complete. The time for dissolution depends on different parameters such as the temperature of calcination, the duration of calcination, the porosity and the particle size of the powder. A minimum of 2 h is necessary to complete dissolution.

15.3.6 Allow the solution to cool, then weigh the hot plate bottle containing the dissolved PuO_2 to the nearest 0.1 mg. Record the weight, m_5. The loss in weight between m_4 and m_5 should be less than 1 %. If it is greater, evaluate possible causes and determine whether additional action is warranted.

15.3.7 Quantitatively transfer the solution to a volumetric flask, using at least four rinses of the hot plate bottle with 0.2 *M* HNO_3, and adjust to volume with 0.2 *M* HNO_3.

15.3.8 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

16. Procedure 8 – Open-Vessel Dissolution (with Reflux Condenser) of Mixed Oxide Powder

16.1 *Apparatus:*

16.1.1 *Hot Plate,* thermostatically controlled, capable of maintaining a surface temperature of up to at least 200°C.

NOTE 19—The surface temperature of a hot plate can vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized before use.

16.1.2 *Perfluoroalkoxy alkane (PFA) polymer narrow-mouth hot plate bottle,* 125 mL.

16.1.3 *Reflux condenser.*

16.2 *Reagents:*

16.2.1 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

16.2.2 *Nitric Acid,* concentrated (sp gr 1.42), 16 *M*.

16.2.3 *Hydrofluoric Acid-Nitric Acid Mixture,* 0.05 *M* HF-14 *M* HNO_3—Add 1.8 mL of 28 *M* HF to 892 mL of 16 *M* HNO_3 and slowly dilute to 1 L with water, adding water slowly to avoid splattering.

16.2.4 *Nitric Acid, 0.2 M*—Add 12.6 mL of 16 *M* HNO_3 to <900 mL of water and dilute to 1 L with water.

16.3 *Procedure:*

16.3.1 Weigh an empty 125 mL PFA polymer narrow-mouth hot plate bottle to the nearest 0.1 mg. Record the weight, m_1.

16.3.2 Transfer 1 to 5 g of sample into the bottle. Weigh the bottle containing the oxide to the nearest 0.1 mg and record the weight m_2. Calculate the weight of sample transferred to the bottle, m_3, by subtracting the tare weight m_1 from m_2.

16.3.3 For each gram of sample, add 4 mL of 0.05 *M* HF—14 *M* HNO_3 mixture.

16.3.4 Weigh the hot plate bottle containing the oxide and acid mixture to the nearest 0.1 mg. Record the weight, m_4.

16.3.5 Set a reflux condenser on the bottle and heat the solution to boiling on a hot plate until dissolution is complete. The time for dissolution depends on different parameters such as the temperature of calcination, the duration of calcination, the porosity and the particle size of the powder. A minimum of 2 h is necessary to complete dissolution.

16.3.6 Allow the solution to cool, then weigh the hot plate bottle containing the dissolved oxide to the nearest 0.1 mg. Record the weight, m_5. The loss in weight between m_4 and m_5 should be less than 1 %. If it is greater, evaluate possible causes and determine whether additional action is warranted.

16.3.7 Quantitatively transfer the solution to a volumetric flask, using at least four rinses of the hot plate bottle with 0.2 *M* HNO_3, and adjust to volume with 0.2 *M* HNO_3.

16.3.8 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

17. Procedure 9 – Closed-Vessel Hot Block Dissolution of Plutonium Oxide Powder

17.1 *Apparatus:*

17.1.1 *Hot Block,* thermostatically controlled, with digestion wells appropriately sized to hold 120 mL digestion vessels (minimum of 16 wells recommended), capable of maintaining a temperature of at least 150°C in the digestion wells.

17.1.2 *Digestion vessels,* 120 mL, polytetrafluoroethylene (PTFE), pressure rated, fitted with pressure relief closures and holders or stands.

NOTE 20—A pressure rating of 172 kPa (25 psi) at 135°C has been found to be satisfactory.

17.1.3 *Centrifuge tubes,* 50 mL, plastic.

NOTE 21—Some centrifuge tubes have been identified as a potential source of iron contamination. Performance testing is recommended prior to use of a particular source of centrifuge tubes to ensure that the tubes do not contaminate the samples.

17.1.4 *Wrench set,* polypropylene, with appropriate sizes for loosening and tightening digestion vessel closures.

17.2 *Reagents:*

17.2.1 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

17.2.2 *Nitric Acid,* concentrated (sp gr 1.42), 16 *M*.

17.2.3 *Hydrofluoric Acid-Nitric Acid Mixture,* 0.1 *M* HF-12 *M* HNO_3—Add 3.6 mL of 28 *M* HF to 764 of 16 *M* HNO_3 and slowly dilute to 1 L with water, adding water slowly to avoid splattering.

17.2.4 *Nitric Acid, 3 M*—Add 191 mL 16 *M* HNO_3 to <800 mL water and dilute to 1 L with water.

17.2.5 *Silicon standard,* 1000 ppm.

17.3 *Procedure:*

17.3.1 Perform the following steps to condition dilution vessels prior to use:

17.3.1.1 Add 10 mL water to each vessel.

17.3.1.2 Cap each vessel and tighten pressure relief closure.

17.3.1.3 Place vessel in hot block well and heat between 120 to 135°C for a minimum of 12 h, then allow to cool to room temperature.

17.3.1.4 Loosen the pressure relief closure, then slowly loosen the screw cap using wrenches as necessary.

17.3.1.5 Dispose of the water.

17.3.1.6 Repeat steps 17.3.1.1 through 17.3.1.5 until caps remain tight after cooling.

17.3.1.7 Rinse each vessel at least three times with water, allow to dry, and store until ready to use.

17.3.2 Prepare a reagent blank (RB) by adding 7 mL of 0.1 *M* HF-12 *M* HNO_3 mixture to a clean, dry, labeled digestion vessel. Carry the RB through the dissolution process described in 17.3.5 through 17.3.15.

17.3.3 Prepare a laboratory control sample (LCS) and duplicate (LCSD) by adding 6.5 mL of 0.1 *M* HF-12 *M* HNO_3 mixture, followed by 0.5 mL of 1000 ppm silicon standard, to a clean, dry, labeled digestion vessel. Carry the LCS and LCSD through the dissolution process described in 17.3.5 through 17.3.15.

17.3.4 For each plutonium oxide sample, perform the following steps:

17.3.4.1 Place a clean, dry, labeled digestion vessel on a balance, and tare the balance.

17.3.4.2 Weigh 0.40 + 0.1 g PuO_2 into the vessel.

17.3.4.3 Add 7 mL of 0.1 *M* HF-12 *M* HNO_3 mixture. If a reaction occurs, evaluate the cause and take appropriate action before proceeding further.

17.3.4.4 Carry each sample through the dissolution process described in 17.3.5 through 17.3.15.

17.3.5 Add approximately 60 mL silicon dioxide to a digestion vessel, and insert a thermometer into the silicon dioxide.

NOTE 22—The vessel containing silicon dioxide is not capped, but is used to monitor temperature during the heating process.

17.3.6 Cap digestion vessels as recommended by the manufacturer, using holder, stand, or wrenches as necessary to a minimum of one-quarter turn past hand tight.

17.3.7 Tighten pressure relief closures.

17.3.8 Place the digestion vessels containing the silicon dioxide, RB, LCS, LCSD, and each sample into hot block wells.

17.3.9 Heat the digestion vessels between 120 to 135°C for a minimum of 24 h, checking temperature every 4 to 6 h to ensure the thermometer is in the range of 120 to 135°C.

17.3.10 Allow vessels to cool to room temperature, then gently tap each vessel on the floor of the glovebox or other containment unit to settle condensation.

17.3.11 Inspect each sample to verify that no solids remain.

17.3.12 Obtain a new, labeled 50 mL centrifuge tube for the RB, LCS, LCSD, and each sample.

17.3.13 Quantitatively transfer each sample (including RB, LCS, and LCSD) into a previously tared and labeled 50 mL centrifuge tube, using at least four rinses of the digestion vessel with approximately 0.75 mL of 0.1 *M* HF-12 *M* HNO_3 mixture. Add sufficient 0.1 *M* HF 12 *M* HNO_3 mixture for a final volume of 10 mL, as marked on the centrifuge tube.

17.3.14 Weigh each of the solutions to the nearest 0.1 mg.

17.3.15 Mix each solution, equilibrate to room temperature, and dispense aliquots for analysis.

18. Procedure 10 – Open-Vessel (with Reflux Condenser) Dissolution of Mixed Oxide Pellets

18.1 *Apparatus:*

18.1.1 *Hot Plate,* thermostatically controlled, capable of maintaining a surface temperature of up to at least 200°C.

NOTE 23—The surface temperature of a hot plate can vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized before use.

18.1.2 *Perfluoroalkoxy alkane (PFA) polymer narrow-mouth hot plate bottle,* 250 mL.

18.1.3 *Reflux condenser.*

18.2 *Reagents:*

18.2.1 *Hydrofluoric Acid,* concentrated (sp gr 1.17), 28 *M*.

18.2.2 *Nitric Acid,* concentrated (sp gr 1.42), 16 *M*.

18.2.3 *Hydrofluoric Acid-Nitric Acid Mixture,* 0.05 *M* HF-14 *M* HNO_3—Add 1.8 mL of 28 *M* HF to 892 mL of 16 *M* HNO_3 and slowly dilute to 1 L with water, adding water slowly to avoid splattering.

18.2.4 *Nitric Acid, 0.2 M*—Add 12.6 mL of 16 *M* HNO_3 to <900 mL of water and dilute to 1 L with water.

18.3 *Procedure:*

18.3.1 Weigh an empty 250 mL PFA polymer narrow-mouth hot plate bottle to the nearest 0.1 mg. Record the weight, m_1.

18.3.2 Add three mixed oxide pellets to the bottle. Weigh the bottle containing the pellets to the nearest 0.1 mg and record the mass, m_2. Calculate the weight of sample transferred to the bottle, m_3, by subtracting the tare weight m_1 from m_2. The mass of the pellets (m_3) should be between 18 to 30 g.

18.3.3 For each gram of sample, add 4 mL of 0.05 *M* HF—14 *M* HNO_3 mixture.

18.3.4 Weigh the hot plate bottle containing the pellets and acid mixture to the nearest 0.1 mg. Record the weight, m_4.

18.3.5 Set a reflux condenser on the bottle and heat the solution to boiling on a hot plate until dissolution is complete. The time for dissolution depends on different parameters related to the pellet manufacturing process. A minimum of 12 h is necessary to complete dissolution.

18.3.6 Allow the solution to cool, then weigh the hot plate bottle containing the dissolved pellets to the nearest 0.1 mg. Record the weight, m_5. The loss in weight between m_4 and m_5 should be less than 1 %. If it is greater, evaluate possible causes and determine whether additional action is warranted.

18.3.7 Quantitatively transfer the solution to a volumetric flask, using at least four rinses of the hot plate bottle with 0.2 M HNO_3, and adjust to volume with 0.2 M HNO_3.

18.3.8 Mix the solution, equilibrate to room temperature, and dispense aliquots for analysis.

19. Reliability

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

19.2 For optimum reliability, every dissolved sample solution must be carefully inspected 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.

19.3 If a residue remains after any of the dissolution procedures, whether this residue is one of the constituents to be measured must be determined. If so, it must be put into solution through further treatments, which are at the discretion of the analyst, and added to the original solution, or the solutions may be analyzed separately.

19.4 If, after multiple treatments to dissolve residues, solids still persist, the originator of the sample should be consulted concerning whether further treatment is required to effect complete solubility. Alternatively, a nondestructive analysis (NDA) can be done on the residue to determine if any component being tested for is still present in the residue. Also, other dissolution treatments described in Refs (**5**, **6**, **7**, **8**, **9**) can be applied.

19.5 If appropriate and sufficiently reliable, the results of the NDA measurement may be applied as a correction to the measurement results on the dissolved sample solution.

20. Quality Control

20.1 Quality control requirements must be considered for both the preparation and dissolution of plutonium materials, and for the subsequent analyses performed on the dissolved aliquots. General guidance for quality control samples can be found in Guide C1210.

20.2 In addition to blanks designed to verify instrumental performance, reagent blanks are used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample preparation.

20.3 It is recommended that at least one instrumental blank, one reagent blank, one LCS and one LCSD be run for each group of dissolutions, but not exceeding 20 samples per group. Although only Procedure 9 (Section 17) includes detailed instructions for blanks, LCS, and LCSD, this recommendation applies to all procedures in this practice.

21. Precision and Bias

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

22. Keywords

22.1 compilation of dissolution techniques; plutonium analysis preparation; plutonium dissolution; plutonium metal dissolution; plutonium oxide dissolution; uranium-plutonium mixed oxide dissolution

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