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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 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:

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

[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 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.

[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.

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 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 H_2O content of less than 10 µL/L and O_2 content less than 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 mg 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 mass 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 mL to 8 mL of 12 *M* HCl and cover with a watch glass.

10.4.4 Allow the metal to dissolve for 10 min 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 mass 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 mg 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 mL 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 mass 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.

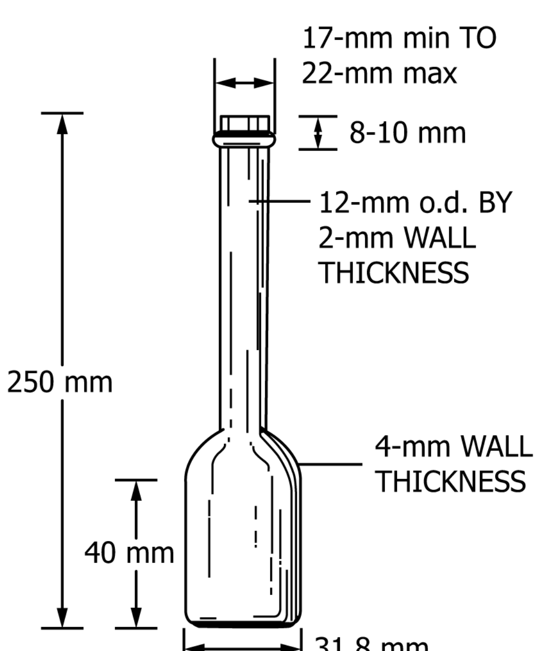

FIG. 1 Sealed-Reflux Dissolution Tube

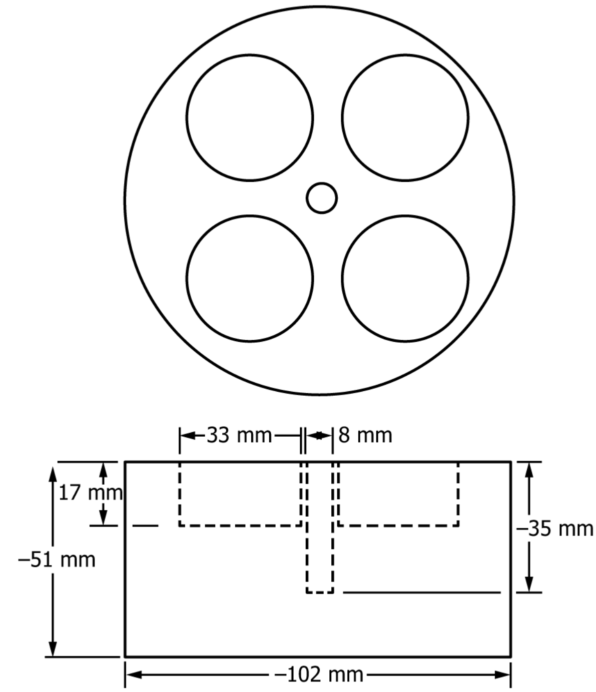

FIG. 2 Heating Block

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.

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 mass 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 mass of sample transferred to the tube by subtracting the mass of the pan from the mass 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 h 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 mass 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 mg to 300 mg sample of plutonium oxide or 1 g 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 mass 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 °C to 625 °C to prevent sample spatter or temperature overshoot, or both, hold at 600 °C to 625 °C for 20 to 30 min. Do not exceed 600 °C 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 mL 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 mass 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 mL 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,