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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Microwave Oven Dissolution of Glass Containing Radioactive and Mixed Wastes[1]

This standard is issued under the fixed designation C 1412; 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 describes a microwave oven practice used to dissolve glass samples that may contain nuclear wastes. The resulting solutions are then used to determine metals and radionuclides in support of glass vitrification plant operations and materials development programs. This practice can be used to dissolve production glass samples, vitrified melter feeds, and sludges.

1.2 This practice is introduced to provide the user with an alternative means to Test Methods C 169 for dissolution of waste containing glass in shielded facilities. Test Methods C 169 is not practical for use in such facilities and with radioactive materials.

1.3 *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:*

C 169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass[2]

C 1109 Test Method for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma—Atomic Emission Spectrometry[3]

C 1111 Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma—Atomic Emission Spectroscopy[3]

C 1285 Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)[3]

C 1317 Practice for Dissolution of Silicate or Acid–Resistant Matrix Samples[3]

C 1342 Practice for Flux Fusion Sample Dissolution[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *product consistency test (PCT)*—a series of test methods as defined in Test Methods C 1285 that evaluate the chemical durability of homogenous and devitrified glasses by measuring the concentrations of chemical species released from a crushed glass to a test solution.

4. Summary of Practice

4.1 The glass samples are ground to a fine powder and digested in a microwave oven using a mixture of hydrofluoric and nitric acids. The sample is then further digested after the addition of hydrochloric acid and boric acid. Boron may be added to the resulting solution to complex fluoride ions and to aid in the dissolution of low–solubility metal fluorides. The solution is then analyzed for metals and radionuclides.

4.2 Boron may interfere with determining certain elements of interest, so the user may process two sample aliquots with one containing no added boron.

5. Significance and Use

5.1 This practice details microwave oven methods to dissolve vitrified feed and product glasses for determining concentrations of metals and radionuclides. Microwave oven dissolution of glass samples as described in this practice is used to dissolve samples for subsequent analysis by plasma spectrometric, atomic absorption, and radiochemical techniques.

5.2 This dissolution method is suitable for dissolving samples of canistered glass containing nuclear wastes with analyte recoveries that are suitable for process control, waste acceptance, and durability testing as described in Refs **1** and **2**.

5.3 The practice will dissolve vitrified melter feed with recovery of analytes satisfactory for glass plant process control.

5.4 This microwave dissolution practice, when used in conjunction with standard practices for alkaline flux fusion of glass (Practices C 1342 and C 1317), can provide solution suitable for determining most metals, radionuclides, and anions of interest.

5.5 The solutions resulting from this practice (after necessary dilutions and preparations) are suitable for analysis by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) as described in Test Methods C 1109 and C 1111, inductively coupled plasma-mass spectrometry (ICP-MS), atomic absorption spectrometry, ion chromatography, and radiochemical methods.

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[1] This practice is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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[2] *Annual Book of ASTM Standards*, Vol 15.02.

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

5.6 This practice can be used to dissolve glass samples for bulk characterizations in support of the PCT as described in Test Methods C 1285.

6. Interferences

6.1 Boron cannot be determined in the solutions obtained from this practice as described in section 4.1 since it may be added to complex excess fluoride ions. Boron may be determined using fusion dissolution as described in Practices C 1342 or C 1317.

6.2 Silicon cannot be determined unless an acid–resistant sample introduction system is used on the ICP-AES or ICP/MS spectrometers. Since Si is the matrix, quantitation is normally not required. However, Si may be measured by fusing the glass using Practices C 1342 or C 1317 and analyzing the resulting solutions.

6.3 Some elements such as Th and the rare earths may not dissolve. An alkaline fusion of the glass using Practices C 1342 or C 1317 may be necessary for quantitative recoveries of these elements.

6.4 Elements that form volatile fluorides may be lost if the microwave digestion vessels vent prior to cooling.

6.5 Low recoveries of Cr, Ni, and Zn may occur due to the addition of boric acid. These elements should be determined in a sample aliquot prior to the addition of the boric acid.

6.6 Incomplete dissolution of some samples may result using the parameters of this practice if the sample is not ground less than 100 mesh.

NOTE 1—The user should determine the recoveries of all elements of analytical interest through comparison of experimental results to values of known materials.

7. Apparatus

7.1 *Laboratory microwave oven* with pressure and temperature control and a digestion vessel capping station.

NOTE 2— A remotely operated microwave oven and capping station may be necessary if shielded operations are required to prevent exposure to sample radiation. Conditions for remote operations may be determined on the bench top/hood and then used to estimate oven parameters for shielded operations without the need for pressure and temperature sensors. Use of microwave sensors in a hot cell may be prohibitive.

7.2 *PTFE microwave digestion vessels* with rupture membranes and capable of operating at greater than 100 psi. Digestion vessel venting and pressure monitoring capability is needed.

7.3 *Analytical balance* capable of weighing to ± 0.1 mg.

7.4 *Polypropylene, polyethylene or PTFE bottles and volumetric flasks* of sufficient quantity and size to meet sample and reagent storage and handling needs.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals must be used for all dissolutions and method blanks. Unless specified, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[4] Other grades may be used, if it is ascertained that the reagent is of sufficiently high purity to permit its use without reducing the accuracy of the determination.

8.2 *Hydrofluoric acid (48 - 51 % w/w)*, concentrated hydrofluoric acid (29 *M* HF).

8.3 *Nitric acid (sp gr 1.42)*, concentrated nitric acid (16 *M* HNO_3).

8.4 *Hydrochloric acid (sp gr 1.18)*, concentrated hydrochloric acid (12 *M* HCl).

8.5 *Boric acid*, reagent grade.

8.6 *Boric acid solution, 0.6 M*, dissolve 37.5 g of boric acid into 1 L of water in a polypropylene bottle.

9. Hazards

9.1 Many of the vitreous feeds and the product glasses from vitrification plants will be radioactive requiring the user of this practice to adhere to site radiation protection practices to avoid exposure to radiation. The microwave dissolution may need to be performed in shielded hoods, glove boxes or hot cells.

9.2 Hydrofluoric acid can cause severe burns upon skin contact that will require special medical attention. Inhalation of HF vapors will cause severe lung damage.

9.3 Microwave digestion vessels operate at high temperature and pressure. The operator must follow all safety precautions for cooling and handling as outlined in the manufacturer's instructions and in–site specific safety guidance.

10. Sample Preparation

10.1 Glass and vitrifier feed samples should be ground to 100 mesh or to a "powdery" consistency prior to weighing into the microwave dissolution vessel. Grinding can be done using an agate mortar and pestle if this introduces no contaminants of interest.

10.2 A tungsten carbide grinding apparatus may also be used and will minimize addition of contaminants of interest to the sample.

11. Procedure

11.1 Tare an aluminum weighing boat or a microwave digestion vessel on the analytical balance.

11.2 Weigh 0.25 ± 0.01 g of the ground sample into the boat or digestion vessel.

NOTE 3—The amount of sample taken can vary depending upon the waste loading of the glass, the analytical sensitivity needed, and the radiation levels encountered. The user of this practice should determine the optimum sample size through experimentation with actual materials.

11.3 Transfer the sample quantitatively to the microwave digestion vessel if a weighing boat was used for the initial sample aliquoting.

11.4 Pipette 5 mL of reagent water into the weighing boat, swirl gently, and then pour into the microwave digestion

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

vessel. Various acids may be used to transfer the contents of the boat to the vessel, but the user must establish potential interference effects.

11.5 Pipette 5 mL of nitric acid and 5 mL of hydrofluoric acid to the microwave digestion vessel and swirl the vessel gently to mix the contents.

11.6 Cap the vessels using the capping station, swirl each vessel to ensure uniform mixing, and then place the vessels symmetrically in the round vessel holder. The use of a capping station is optional.

11.7 Follow laboratory and manufacturer's operating directions for loading the vessels and connecting the temperature and pressure indicators and for shielded facility operations.

11.8 Microwave the samples at 100 psi for 15 min.

11.9 Cool the vessels in an ice bath for at least 30 min to ensure ambient pressure. Vent the vessels following established laboratory operating practice.

NOTE 4—The microwave vessels and contents must be cool to ambient temperature prior to uncapping or the cap will blow off violently expelling the contents.

11.10 Add 5 mL of concentrated hydrochloric acid and 40 mL of the 0.6 *M* boric acid solution to each vessel.

11.11 Reserve an aliquot for analysis without the addition of boric acid for determination of metals subject to low recoveries in the presence of boron.

11.12 Recap the vessels, place them in the holder, reconnect vent tubes and monitoring sensors (if used).

11.13 Redigest the samples at 80 psi for an additional 30 min.

11.14 After cooling, uncap the vessels and transfer the contents of the vessels to a 200 mL PTFE volumetric flask and make to volume with water.

NOTE 5—If internal standards such as Sc are desired for ICP-ES analysis or if isotopic mass standards for ICP-MS are desired, then add these elements to the sample flasks at the appropriate concentration prior to diluting to final volume.

11.15 A method blank should be prepared by adding all reagents to a digestion vessel and carrying the solution through the entire process. Also prepare a duplicate and matrix spike sample for QA parameter determination.

12. Precision and Bias

12.1 This practice addresses only the preparation steps in the overall preparation and measurement of analytes in nuclear waste containing glass and thus does not produce any measurements. Hence a statement of precision and bias is not meaningful.

12.2 Data obtained from round-robin glass samples using this dissolution method and subsequent analysis by ICP-ES, AA, and radiochemical methods are reported in Refs **3** and **4**.

13. Keywords

13.1 ICP analysis; microwave digestion; nuclear waste; vitrified glass

REFERENCES

(**1**) *Waste Acceptance Product Specifications for Vitrified High-Level Waste Forms*, DOE-DWPD-FY 93-0288.

(**2**) Bibler, N.E. and Jantzen, C.M., *The Product Consistency Test And Its Role in The Waste Acceptance Process for DWPF Glass*, Proceedings of Waste Management 89, Vol. I, Roy G. Post, ed.

(**3**) *Product Consistency Test Round Robin Conducted by the Materials Characterization Center-Summary Report*. USDOE Report PNL P 6967, Battelle Pacific Northwest Laboratory, Richland, WA, September 1989.

(**4**) *Nuclear Waste Analytical Round Robins 1-6, Summary Report*, G.L. Smith and S.C. Marschman, Pacific Northwest Lab, 1993.