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Standard Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis[1]

This standard is issued under the fixed designation C1463; 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 These practices cover techniques suitable for dissolving glass samples that may contain nuclear wastes. These techniques used together or independently will produce solutions that can be analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES), inductively coupled plasma mass spectrometry (ICP-MS), atomic absorption spectrometry (AAS), radiochemical methods and wet chemical techniques for major components, minor components and radionuclides.

1.2 One of the fusion practices and the microwave practice can be used in hot cells and shielded hoods after modification to meet local operational requirements.

1.3 The user of these practices must follow radiation protection guidelines in place for their specific laboratories.

1.4 Additional information relating to safety is included in the text.

1.5 The dissolution techniques described in these practices can be used for quality control of the feed materials and the product of plants vitrifying nuclear waste materials in glass.

1.6 These practices are introduced to provide the user with an alternative means to Test Methods C169 for dissolution of waste containing glass in shielded facilities. Test Methods C169 is not practical for use in such facilities and with radioactive materials.

1.7 The ICP-AES methods in Test Methods C1109 and C1111 can be used to analyze the dissolved sample with additional sample preparation as necessary and with matrix effect considerations. Additional information as to other analytical methods can be found in Test Method C169.

1.8 Solutions from this practice may be suitable for analysis using ICP-MS after establishing laboratory performance criteria.

1.9 *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.* Specific precautionary statements are given in Section 18.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass

C1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy

C1111 Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectroscopy

C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste

C1285 Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)

D1193 Specification for Reagent Water

3. Summary of Practice

3.1 The three practices for dissolving silicate matrix samples each require the sample to be dried and ground to a fine powder.

3.2 In the first practice, a mixture of sodium tetraborate ($Na_2B_4O_7$) and sodium carbonate (Na_2CO_3) is mixed with the sample and fused in a muffle for 25 min at 950°C. The sample is cooled, dissolved in hydrochloric acid, and diluted to appropriate volume for analyses.

3.3 The second practice described in this standard involves fusion of the sample with potassium hydroxide (KOH) or sodium peroxide (Na_2O_2) using an electric bunsen burner,

[1] These practices are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.05 on Methods of Test.

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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.

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dissolving the fused sample in water and dilute HCl, and making to volume for analysis.

3.4 Dissolution of the sample using a microwave oven is described in the third practice. The ground sample is digested in a microwave oven using a mixture of hydrofluoric (HF) and nitric (HNO_3) acids. Boric acid is added to the resulting solution to complex excess fluoride ions.

3.5 These three practices offer alternative dissolution methods for a total analysis of a glass sample for major, minor, and radionuclide components.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[3]

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean at least Type II reagent water in conformance with Specification D1193.

PRACTICE 1—FUSION WITH SODIUM TETRABORATE AND SODIUM CARBONATE

5. Scope

5.1 This practice covers flux fusion sample decomposition and dissolution for the determination of SiO_2 and many other oxides in glasses, ceramics, and raw materials. The solutions are analyzed by atomic spectroscopy methods. Analyte concentrations ranging from trace to major levels can be measured in these solutions, depending on the sample weights and dilution volumes used during preparation.

6. Technical Precautions

6.1 This procedure is not useful for the determination of boron or sodium since these elements are contained in the flux material.

6.2 The user is cautioned that with analysis by ICP-AES, AAS, and ICP-MS, the high sodium concentrations from the flux may cause interferences.

6.3 Elements that form volatile species under these alkaline fusion conditions may be lost during the fusion process (that is, As and Sb).

7. Apparatus

7.1 *Platinum Crucibles,* 30 mL.

7.2 *Balance,* analytical type, precision to 0.1 mg.

7.3 *Furnace,* with heating capacity to 1000°C.

7.4 *Crucible Tongs,* (cannot be made of iron, unless using platinum-clad tips).

7.5 *Polytetrafluoroethylene (PTFE) Beaker,* 125-mL capacity.

7.6 *Magnetic Stir Bar,* PTFE-coated (0.32 to 0.64 cm).

7.7 *Magnetic Stirrer.*

7.8 *Mortar and Pestle,* agate or alumina (or equivalent grinding apparatus).

7.9 *Sieves,* 100 mesh.

8. Reagents and Materials

8.1 *Anhydrous Sodium Carbonate* (Na_2CO_3).

8.2 *Anhydrous Sodium Tetraborate* ($Na_2B_4O_7$).

8.3 *Sodium Nitrate* ($NaNO_3$).

8.4 *Hydrochloric Acid* (HCl), 50 % (v/v), made from concentrated hydrochloric acid (sp gr 1.19) and water.

8.5 *Nitric Acid* (HNO_3), 50 % (v/v), made from concentrated nitric acid (sp gr 1.44) and water.

9. Hazards and Precautions

9.1 Follow established laboratory practices when conducting this procedure.

9.2 The operator should wear suitable protective gear when handling chemicals.

9.3 The dilution of concentrated acids is conducted in fume hoods by cautiously adding an equal part acid to an equal part of deionized water slowly and with constant stirring.

9.4 Samples that are known or suspected to contain radioactive materials must be handled with the appropriate radiation control and protection as prescribed by site health physics and radiation protection policies.

9.5 Samples that are known or suspected to contain toxic, hazardous, or radioactive materials must be handled to minimize or eliminate employee exposure. Fusion and leaching of the fused samples must be performed in a fume hood, radiation-shielded facility, or other appropriate containment.

10. Sample Preparation

10.1 If the material to be analyzed is not in powder form, it should first be broken into small pieces by placing the sample in a plastic bag and then striking the sample with a hammer. The sample should then be ground to pass a 100-mesh sieve using a clean mortar and pestle such as agate or alumina

11. Procedure

11.1 Weigh 50 to 250 mg of a powdered sample into a platinum crucible on an analytical balance to ±0.1 mg. The sample size is dependent on the analyte concentration.

NOTE 1—Although the larger sample size has generally worked well, some matrices may not dissolve entirely. Try smaller sample sizes if that is the case.

11.2 Add 0.5 ± 0.005 g each of Na_2CO_3 and $Na_2B_4O_7$ to the crucible containing the sample.

11.3 Stir the sample/flux mixture in the crucible with a spatula until a mixture is obtained. Prepare a reagent blank

[3] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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11.4 For samples containing minor to major elements that do not oxidize readily (such as Pb, Fe, etc.), add 300 mg of sodium nitrate. If desired, a Pt lid can be placed on the crucible to reduce splattering. When adding nitrate, 50 % v/v HNO_3 should be the diluting acid in order to reduce the attack on platinum in 11.6.

11.5 Using the crucible tongs, place the crucible containing the sample/flux mixture into a muffle furnace for 25 min at a temperature of 950°C. Remove the crucible from the furnace and allow the melt to cool to room temperature.

11.6 Place a stir bar in each crucible and add 4 mL 50 % v/v HCl, and then dilute with H_2O to near the top of the crucible.

NOTE 2—In some cases, 50 % v/v HNO_3 may be more appropriate than HCl (that is, samples for ICP-MS, high lead samples, or when sodium nitrate was added).

11.7 Place the crucible on the magnetic stirrer, and stir until the sample melt is dissolved completely (approximately 30 min). If undissolved material remains, the fusions described in Section 20 may need to be tried for cross correlation.

11.8 To a calibrated volumetric flask, typically 100, 250, 500, or 1000 mL, add enough 1:1 HCl to make the final concentration 2 % (including the acid already in the crucible). The final volume is determined by the expected analyte concentrations. Quantitatively transfer the sample solution, and dilute.

11.9 The dilution volume is determined by the user of the practice and is dependent upon the desired analysis.

11.10 See Appendix X1 for examples of analytical data using solutions from this fusion.

PRACTICE 2—FUSION WITH POTASSIUM HYDROXIDE OR SODIUM PEROXIDE

12. Scope

12.1 This practice covers alkaline fusion of silicate matrix samples (or other matrices difficult to dissolve in acids) using an electric Bunsen burner mounted on an orbital shaker. This practice has been used successfully to dissolve borosilicate glass, dried glass melter feeds, various simulated nuclear waste forms, and dried soil samples.

12.2 This fusion apparatus and the alkaline fluxes described are suitable for use in shielded radiation containment facilities such as hot cells and shielded hoods.

12.3 When samples dissolved using this practice are radioactive, the user must follow radiation protection guidelines in place for such materials.

13. Summary of Practice

13.1 An aliquot of the dried and ignited sample is weighed into a tared nickel or zirconium metal crucible and an appropriate amount of alkaline flux (potassium hydroxide or sodium peroxide) is added. The crucible is placed on a preheated electric Bunsen burner (1000°C capability) mounted on an orbital shaker. The speed of the shaker is adjusted so that the liquefied alkali metal flux and the sample are completely fused at the bottom of the crucible. When the fusion is complete (about 5 min), the crucible is removed from the heater and cooled to room temperature. The fused mixture is dissolved in water, acidified with hydrochloric acid, and diluted to an appropriate volume for subsequent analysis.

13.2 With appropriate sample preparation, the solution resulting from this procedure can be analyzed for trace metals by ICP-AES, ICP-MS, and AAS, and for radionuclides using applicable radiochemical methods.

14. Significance and Use

14.1 This practice describes a method to fuse and dissolve silicate and refractory matrix samples for subsequent analysis for trace metals and radionuclides. These samples may contain high-level radioactive nuclear waste. Nuclear waste glass vitrification plant feeds and product can be characterized using this dissolution method followed by the appropriate analysis of the resulting solutions. Other matrices such as soil and sediment samples and geological samples may be totally dissolved using this practice.

14.2 This practice has been used to analyze round-robin simulated nuclear waste glass samples.

14.3 This practice can be used for bulk analysis of glass samples for the product consistency test (PCT) as described in Test Methods C1285 and for the analysis of monolithic radioactive waste glass used in the static leach test as described in Test Method C1220.

14.4 This practice can be used to dissolve the glass reference and testing materials described in Refs (**1**) and (**2**).[4]

15. Interferences

15.1 Elements that form volatile species under these alkaline fusion conditions will be lost during the fusion process.

15.2 The high alkali metal (Na or K) content of the resulting sample solutions can cause interference with ICP nebulizer and torch assemblies due to salt deposition. Dilution of the sample solutions may be necessary.

15.3 The metallic impurities, that is, Na, K, in the alkaline flux used to fuse the samples can cause a positive bias if proper corrections are not applied. Method blanks must be determined to allow correction for flux impurity concentration.

16. Apparatus

16.1 *Analytical Balance,* capable of weighing to ± 0.1 mg.

16.2 *Electric Bunsen Burner,* capable of heating to 1000°C.[5] to accommodate the larger size (100 mL nickel) metal crucibles, the heat shield on top of the electric Bunsen Burner is wrapped with a noncorrosive wire such as inconel at three evenly distributed locations. With the wire on the heat shield, the large size crucibles are better supported and more easily removed. A wire basket made from the noncorrosive wire is also fabricated so that smaller size crucibles (55 mL zirconium)

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

[5] Electric bunsen burners are available from most major laboratory supply houses.

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