



Standard Practice for Dissolution of Silicate or Acid-Resistant Matrix Samples¹

This standard is issued under the fixed designation C 1317; 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 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.

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

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

1.4 *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 8.

2. Referenced Documents

2.1 ASTM Standards:

C 169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass²

C 1109 Test Method for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectrometry³

C 1111 Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectroscopy²

C 1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste²

C 1285 Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)²

D 1193 Specification for Reagent Water⁴

3. Summary of Practice

3.1 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.

3.2 With appropriate sample preparation, the solution resulting from this procedure can be analyzed for trace metals by inductively coupled plasma-atomic emission spectroscopy (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), and atomic absorption spectroscopy, and for radionuclides using applicable radiochemical methods.

4. Significance and Use

4.1 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. The ICP-AES methods in Test Methods C 1109 and C 1111 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 C 169.

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

4.3 This practice can be used for bulk analysis of glass samples for the product consistency test (PCT) as described in Test Methods C 1285 and for the analysis of monolithic radioactive waste glass used in the static leach test as described in Test Method C 1220.

4.4 This practice can be used to dissolve the glass reference and testing materials described in Refs (1) and (2).⁵

5. Interferences

5.1 Elements that form volatile species under these alkaline

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. Current edition approved Nov. 10, 1995. Published March 1996.

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

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

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ The boldface numbers in parentheses refer to the list of references at the end of this practice.

fusion conditions will be lost during the fusion process.

5.2 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.

5.3 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.

6. Apparatus

6.1 *Analytical Balance*, capable of weighing to ± 0.0001 g.

6.2 *Electric Bunsen Burner*, capable of heating to 1000°C .⁶ 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 inonel 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) that pass through the heat shield are supported evenly in the heating mandrel of the electric Bunsen burner. Fig. 1 shows the electric Bunsen burner mounted on the orbital shaker with the above modifications for crucible mounting.

6.3 *Orbital Shaker*, including a holder fabricated to fasten the electric Bunsen burner on the platform (see Fig. 1).⁷

6.4 *Manual Adjustable Power Supply*, for controlling the

temperature of the electric Bunsen burner.⁸

6.5 *Zirconium Metal Crucible*, 55 mL capacity, high form. Different shape and capacity crucibles also may be used when necessary.

6.6 *Nickel Metal Crucible*, 100 mL capacity, high form. Different shape and capacity crucibles also may be used when necessary.

6.7 *Hot Plate or Steam Bath*, capable of heating to 100°C .

7. Reagents and Materials

7.1 *Purity of Reagents*—All chemicals used in this practice are to be reagent grade. Unless otherwise indicated all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.⁹

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean at least Type II reagent water conforming to Specification D 1193.

7.3 *Potassium Hydroxide* (KOH)—pellet.

7.4 *Potassium Nitrate* (KNO_3)—crystal.

7.5 *Sodium Peroxide* (Na_2O_2)—granular.

7.6 *Hydrochloric Acid* (HCl)—concentrated, sp gr 1.19.

7.7 *Nitric Acid Solution* (2 Vol. %)—Add 20 mL of concentrated nitric acid (HNO_3 , sp gr 1.42) to 950 mL of water while stirring. Make to 1 L volume and store in a polyethylene bottle.

7.8 *Oxalic Acid*—crystals.

⁸ The Model 01575-26 power supply available from Cole-Parmer Instrument Company has been found to be suitable.

⁹ *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.

⁶ Electric bunsen burners are available from most major laboratory supply houses.

⁷ Orbital shaker, Model 04732-00 available from Cole-Parmer Instrument Company, has been found to be suitable.



FIG. 1 Electric Bunsen Burner Mounted on the Orbital Shaker