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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for
Dissolution of Silicate or Acid-Resistant Matrix Samples[1]

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 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[2]

C 1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste[2]

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

D 1193 Specification for Reagent Water[4]

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

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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. 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 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) 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.

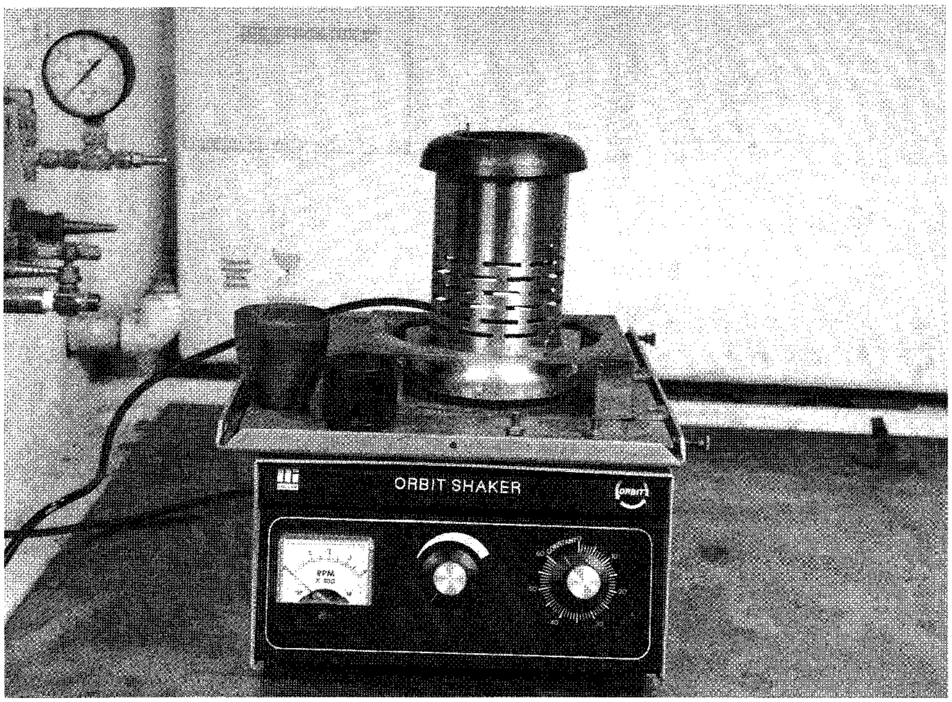

FIG. 1 Electric Bunsen Burner Mounted on the Orbital Shaker

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[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.

[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.

8. Hazards and Precautions

8.1 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.

8.2 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. Personal protective equipment must be worn when appropriate. All site good laboratory safety and industrial hygiene practices must be followed.

8.3 Sodium peroxide is a strong oxidizer. Precaution must be taken when fusions are performed on samples containing materials that are readily oxidized.

8.4 Samples containing significant concentrations of phosphates (greater than 5 %) cannot be fused in a zirconium metal crucible using sodium peroxide. The phosphate destroys the oxide layer on the crucible, resulting in severe corrosion. Aluminum oxide crucibles can be substituted for fusion of samples containing phosphates greater than 5 %.

9. Sample Preparation

9.1 *Wet or Slurry Samples*:

9.1.1 Dry wet or slurry samples in a tared porcelain crucible at 105°C. Grind the dried sample in a porcelain mortar to a particle size to pass a No. 200 (74 µm) sieve.

9.1.2 Weigh a portion (approximately 3 g) of the dried and ground sample described in 9.1.1 to the nearest 0.001 g in a tared porcelain crucible. Ignite the sample at 1000°C and determine the sample loss on ignition factor (I_F), where:

$$I_F = \frac{W_i - W_f}{W_i} \tag{1}$$

where:

W_i = initial sample weight, and

W_f = sample weight after ignition.

9.2 *Dry Solid or Oxide Samples*:

9.2.1 Grind the dry solid or oxide sample to a particle size to pass a No. 200 (74 µm) sieve.

9.2.2 Weigh a portion (approximately 3 g) of the ground sample described in 9.2.1 to the nearest 0.001 g in a tared porcelain crucible. Ignite the sample at 1000°C and determine the ignition factor in accordance with Eq 1.

NOTE 1—The loss on ignition for dry solid or oxide samples may be negligible.

10. Procedure

10.1 *Potassium Hydroxide Fusion*—The KOH fusion is performed in a nickel metal crucible.

10.1.1 The choice of fusion methods described in 10.1 and 10.2 is determined by the analyte elements to be determined; that is, if combinations of Na, K, Ni, or Zr are to be determined, then one or both of the fusion methods may have to be performed.

10.1.2 Set the manually adjustable power controller that supplies power to the electric Bunsen burner so that 1.6 g of Na_2O_2 in a zirconium crucible will melt within 1 to 2 min.

10.1.3 Tare a nickel metal crucible to the nearest 0.001 g.

10.1.4 Weigh an aliquot of the ground sample described in 9.1.1 or 9.2.1, which is equivalent to 0.350 ± 0.050 g of ignited sample (9.1.2 or 9.2.2). Determine the amount of dried sample (W_s) to be aliquoted by using the ignition factor from 9.1.2 as follows:

$$W_s = \frac{0.350\ g}{1 - I_F} \tag{2}$$

10.1.5 Add 1.600 ± 0.2 g of KOH pellets. Record the weight of KOH added to the crucible to the nearest 0.001 g. Swirl the crucible to mix the sample and the KOH pellets completely.

10.1.6 Reagent grade KOH will contain trace amounts of sodium as an impurity. A correction for this flux impurity should be made to the sodium found in the sample.

10.1.7 Set the crucible on the preheated electric Bunsen burner and turn on the orbital shaker.

10.1.8 Fuse the sample mixture for approximately 5 min or until the fusion is complete. If at the completion of the fusion or after about 5 min of heating, there is still undissolved material, remove the crucible from the burner, allow to cool, and add 0.5 mL of water. Replace the crucible on the burner and continue fusion until dissolution is complete.

NOTE 2—During the KOH fusion, the flux will become more viscous as the fusion continues. If the temperature of the electric Bunsen burner is set too high, the KOH will solidify before the fusion is complete. Once the fusion mixture has solidified and the heating is continued, further dissolution of the sample ceases and some of the dissolved silicates in the sample will dehydrate, resulting in incomplete dissolution of the fused sample.

10.1.9 When fusion is complete, remove the crucible from the burner and allow to cool to room temperature.

10.1.10 Add water drop-wise to the crucible until the initial vigorous reaction subsides. Add a total of about 10 mL of water to dissolve the fused mixture. Transfer the solution to a 250-mL volumetric flask. If the initial dissolution was not complete, continue to add water until all the fused sample has been dissolved and then transfer the resulting solution to the flask.

10.1.11 Add 50 mL of 1 + 1 HCl and 0.5 g of oxalic acid to the volumetric flask. Dilute with water until the volume in the flask is about 150 mL. If the solution is still cloudy (white precipitate), heat the flask carefully on a hot plate to near boiling. Continue to heat without boiling until the precipitate dissolves. Cool the flask to room temperature and make the solution to volume with water. Mix the solution thoroughly.

NOTE 3—Oxalate in an acidic solution will dissolve zirconium phosphate. Heating accelerates the dissolution rate. If dehydrated silicic acid was produced during the fusion, this material will not dissolve and the fusion process (10.1.8) will need to be repeated.

10.1.12 A ten-fold dilution of this solution in 2 % nitric acid is necessary for ICP-AES or atomic absorption analysis for metals.

10.2 *Sodium Peroxide Fusion*—The Na_2O_2 fusion is performed in a zirconium metal crucible.

10.2.1 Set the adjustable power controller on the electric Bunsen burner so that 1.6 g of Na_2O_2 in a zirconium crucible will melt in 1 to 2 min. This is the same setting determined in 10.1.2.

10.2.2 Tare a zirconium crucible to within 0.001 g.

10.2.3 Weigh an aliquot of the ground sample described in 9.1.1 or 9.2.2, which is equivalent to 0.350 ± 0.050 g of ignited sample. Use Eq 2 to calculate the aliquot of the dried sample to fuse.

10.2.4 Add 1.600 ± 0.2 g of granular Na_2O_2. Record the weight of Na_2O_2 added to the nearest 0.001 g. Swirl the crucible to completely mix the sample into the Na_2O_2 granules.

10.2.5 Set the crucible on the preheated electric Bunsen burner and turn on the orbital shaker. Fuse the mixture for approximately 5 min or until fusion is complete.

10.2.6 Remove the crucible from the burner and cool to room temperature.

10.2.7 Add water drop-wise until the initial vigorous reaction subsides. Add about 10 mL of water to dissolve the fusion mixture. Transfer the solution to a 250-mL volumetric flask. If the initial dissolution was not complete, continue to add water, and add the solution to the flask.

10.2.8 Add 50 mL of 1 + 1 HCl and 0.5 g of oxalic acid to the volumetric flask. Dilute with water until the volume in the flask is about 150 mL. If the solution is cloudy (white precipitate) carefully heat the flask on a hot plate to near boiling. Continue heating the flask without boiling until the precipitate dissolves. Refer to Note 3 if the precipitate will not dissolve.

10.2.9 Cool the solution to room temperature, make to 250 mL, and mix thoroughly.

10.2.10 A ten-fold dilution of this solution in 2 % nitric acid is necessary for ICP-AES or atomic absorption analysis for metals.

11. Precision and Bias

11.1 This practice addresses only the preparation steps in the overall preparation and measurement of the sample analytes. Since the preparation alone does not produce any results, the user must determine the precision and bias resulting from this preparation and subsequent analysis.

12. Keywords

12.1 alkali fusion; glass dissolution; high-level nuclear waste; silicate matrix; waste vitrification

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE ANALYSES OF GLASS USING THIS FUSION PRACTICE

X1.1 Tables 1-4 are included to demonstrate analytical recoveries experienced by a single laboratory using this fusion dissolution practice followed by ICP-AES analysis of the dissolved samples. The reference glasses used are: Table 1—EA reference glass values were established by the manufacturer; Table 2—Analytical Reference Glass-1 (ARG-1) reference values were established by the manufacturer; Table 3 and Table 4—The reference values are from National Institutes of Standards and Technology (NIST) certificates for the glass SRMs used. The EA and ARG-1 glasses were special production and not generally available.

TABLE 1 Results from Analysis of EA Reference Glass

	Weight % Oxide		Difference	Percent Difference
	Determined Value	Reference Value		
Al_2O_3	3.71	3.70	0.01	0.27
B_2O_3	11.0	11.28	−0.28	−2.48
CaO	1.17	1.12	0.05	4.46
Fe_2O_3	9.48	7.38	2.10	28.46
La_2O_3	0.42	0.42	0.00	0.00
K_2O	[A]	0.04	...	...
Li_2O	4.27	4.26	0.01	0.23
MgO	1.73	1.72	0.01	0.58
MnO	1.35	1.34	0.01	0.75
Na_2O	14.3	16.8	−2.50	−14.88
NiO	0.59	0.57	0.02	3.51
SiO_2	49.6	48.7	0.90	1.85
TiO_2	0.70	0.70	0.00	0.00
ZrO_2	0.47	0.46	0.01	2.17

[A] Potassium is not reported on ICP data.

TABLE 2 Results from Analysis of Analytical Reference Glass-1

	Weight % Oxide		Difference	Percent Difference
	Determined Value	Reference Value		
Al_2O_3	4.69	4.73	−0.04	−0.85
B_2O_3	8.35	8.67	−0.32	−3.69
BaO	0.087	0.088	−0.001	−1.14
CaO	1.50	1.53	−0.03	−1.96
Cr_2O_3	0.10	0.093	0.007	7.53
CuO	not detected	0.004	...	...
Fe_2O_3	14.6	14	0.6	4.29
K_2O	[A]	2.71	...	...
Li_2O	3.20	3.21	−0.01	−0.31
MgO	0.86	0.86	0	0.00
MnO_2	2.27	2.31	−0.04	−1.73
Na_2O	11.2	11.5	−0.3	−2.61
NiO	1.06	1.05	0.01	0.95
P_2O_5	0.36	0.25	0.11	44.00
SiO_2	49.1	47.9	1.2	2.51
SrO	0.005	0.0037	0.0013	35.14
TiO_2	1.15	1.15	0	0.00
ZnO	0.02	0.02	0	0.00
ZrO_2	0.14	0.13	0.01	7.69

[A] Potassium is not reported on ICP data.

X1.2 To determine the suite of elements on Tables 1-4, both the KOH and Na_2O_2 fusions were used to dissolve each of the standard glasses. With the following exceptions, the results in the tables are an average from both fusions:

Na_2O concentrations are from the KOH fusion only,
NiO concentrations are from the Na_2O_2 fusion only, and
ZrO_2 concentrations are from the KOH fusion only.

X1.3 Analytical results for K_2O are not presented since ICP-AES does not have a sufficient lower quantitation limit for

TABLE 3 Results from Analysis of SRM 1411 Borosilicate Glass

	Weight % Oxide		Difference	Percent Difference
	Determined Value	SRM 1411 Value		
Al_2O_3	5.61	5.68	−0.07	−1.17
B_2O_3	10.57	10.94	−0.37	−3.41
BaO	4.82	5.00	−0.18	−3.60
CaO	2.13	2.18	−0.05	−2.14
Fe_2O_3	0.11	0.05	0.06	120.00
K_2O	[A]	2.97	...	...
MgO	0.36	0.33	0.03	8.08
Na_2O	9.85	10.14	−0.29	−2.86
SiO_2	55.49	58.01	−2.52	−4.35
ZnO	3.95	3.85	0.10	2.60

[A] Potassium is not reported on ICP data.

TABLE 4 Results from Analysis of SRM 1412 Borosilicate Glass

	Weight % Oxide		Difference	Percent Difference
	Determined Value	SRM 1412 Value		
Al_2O_3	7.42	7.52	−0.10	−1.33
B_2O_3	4.39	4.53	−0.14	−3.09
BaO	4.44	4.67	−0.23	−4.93
CaO	4.55	4.53	0.02	0.44
CdO	4.31	4.38	−0.08	−1.71
Fe_2O_3	0.06	0.031	0.02	77.42
K_2O	[A]	4.14	...	...
Li_2O	4.34	4.5	−0.17	−3.67
MgO	4.45	4.69	−0.25	...
Na_2O	4.82	4.69	0.13	2.77
PbO	4.46	4.4	0.06	1.36
SiO_2	40.25	42.38	−2.14	−5.04
ZnO	4.45	4.48	−0.04	−0.78

[A] Potassium is not reported on ICP data.

this element. Potassium can be determined from a Na_2O_2 fusion of the glass sample followed by atomic absorption spectroscopy analyses.

REFERENCES

(**1**) Jantzen, C.M., Bibler, N.E., Beam, D.C., Crawford, C.L., and Pickett, M.A., "Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material," Report WSRC-TR-346, Rev. 1, Westinghouse Savannah River Co., Aiken, SC, June 1993.

(**2**) Mellinger, G.B., and Daniel, J.L., "Approved Reference and Testing Materials for Use in Nuclear Waste Management Research and Development Programs," Report PNL-4955-2, Pacific Northwest Laboratory, Richland, WA, December 1984.