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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Flux Fusion Sample Dissolution[1]

1. Scope

1.1 This practice covers flux fusion sample decomposition and dissolution for the determination of SiO_2 as well as 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.

1.2 *This standard does not purport to address all 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 1109 Test Method for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectrometry[2]

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]

C 1317 Practice for Dissolution of Silicate or Acid Resistant Matrix Samples[2]

D 1193 Specifications for Reagent Water[3]

3. Summary of Practice

3.1 A ground sample is weighed into a platinum crucible, and an appropriate amount of alkaline flux (Na_2CO_3 and $Na_2B_4O_7$) is added. The mixture is fused in a muffle furnace (950°C for 25 min), cooled to room temperature, dissolved 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 flame atomic absorption spectroscopy.

4. Significance and Use

4.1 This practice describes a method for fusing and dissolving silicate and refractory matrix samples for subsequent analysis for trace metals and radionuclides. Glass vitrification plant feeds and product can be characterized using this dissolution method, followed by the appropriate analysis of the resulting solutions as described in Test Methods C 1109 and C 1111.

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 as described in Test Methods C 1220 and C 1285.

4.4 This practice is a companion to Practice C 1317. These procedures can be used to dissolve borosilicate glasses containing nuclear wastes for subsequent analysis.

5. Technical Precautions

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

5.2 The user is cautioned that with analysis by ICP emission, flame atomic absorption, and ICP-MS, the high sodium concentrations from the flux may cause interferences.

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

6. Apparatus

6.1 *Platinum Crucibles*, 30 mL.

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

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

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

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

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

6.7 *Magnetic Stirrer*.

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

6.9 *Sieves*, 100 mesh.

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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 12.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

7. Reagents and Materials

7.1 *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.[4]

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

7.3 *Anhydrous Sodium Carbonate* (Na_2CO_3).

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

7.5 *Sodium Nitrate* ($NaNO_3$).

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

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

8. Hazards and Precautions

8.1 Follow established laboratory practices when conducting this procedure.

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

8.3 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. Sample Preparation

9.1 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.

10. Procedure

10.1 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.

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

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

10.4 For samples containing minor to major elements that do not oxidize readily (such as Pb, Fe, etc.), add 300 mg of a 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 Step 10.6.

10.5 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.

10.6 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).

10.7 Place the crucible on the magnetic stirrer, and stir until the sample melt is dissolved completely (approximately 30 min). If undissolved material remains, a companion practice (Practice C 1317) may need to be tried for cross correlation.

10.8 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. Keywords

11.1 borosilicate glass; dissolution; fusion

APPENDIX

(Nonmandatory Information)

X1. Expected Precision and Bias

X1.1 This procedure 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.

X1.2 The data given in Tables X1.1-X1.3 provide an indication of expected precision and bias when using this dissolution procedure to analyze standard reference glasses. These data were obtained by analyzing aliquots of the dissolved sample using ICP-AES. Tables X1.1 and X1.3 show the known target composition, mean weight percent found using this dissolution, and standard deviation and percent relative standard deviation (RSD).

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[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE X1.1 Fusion Dissolution, ARG Glass

N = 36[A]

Oxide	Target[B] Wt %	Mean Wt %	Standard Deviation	% RSD
Al_2O_3	4.53	4.73	0.022	0.5
Cr_2O_3	0.096	0.093	0.001	0.8
SiO_2	48.6	47.9	0.157	0.3
TiO_2	1.12	1.15	0.007	0.6

[A] Six samples of the same glass were ground independently. Each sample was dissolved in triplicate, and each dissolution was analyzed on the ICP in duplicate.

[B] Target composition.

TABLE X1.2 Fusion Dissolution, WVRG-6 Glass

N = 6[A]

Oxide	Target[B] Wt %	Mean Wt %	Standard Deviation	% RSD
Al_2O_3	6.00	6.02	0.052	0.9
BaO	0.16	0.14	0.004	3.0
CaO	0.48	0.57	0.013	2.3
CoO	0.02	0.019	0.001	3.0
Cr_2O_3	0.14	0.17	0.005	3.3
CuO	0.03	0.055	0.002	3.9
Fe_2O_3	12.02	11.9	0.207	1.8
MgO	0.89	0.92	0.021	2.2
MnO_2	1.01	1.09	0.021	1.9
SiO_2	40.98	40.9	0.283	0.7
SrO	0.02	0.024	0.000	0.0
TiO_2	0.80	0.80	0.019	2.4
ZnO	0.02	0.031	0.001	2.0
ZrO_2	1.32	1.30	0.035	2.7

[A] Three samples of the same glass were ground independently. Each sample was dissolved in duplicate and analyzed on the ICP.

[B] Target composition.

TABLE X1.3 Fusion Dissolution, NIST Standard 93a

N = 2[A]

Oxide	Target[B] Wt %	Mean Wt %	Standard Deviation	% RSD
Al_2O_3	2.2(8)	2.29	0.021	0.9
SiO_2	80.8	80.4	0.071	0.1

[A] The sample was ground, dissolved in duplicate, and analyzed by ICP.

[B] National Institute for Standards and Technology supplied data. The numbers in parentheses are for information only and are not considered significant.