



Standard Practice for Flux Fusion Sample Dissolution¹

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1. Scope

1.1 This practice covers flux fusion sample decomposition and dissolution for the determination of SiO₂ 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 Spectrometry²
- C 1111 Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectrometry²
- 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
- C 1317 Practice for Dissolution of Silicate or Acid Resistant Matrix Samples²
- D 1193 Specifications for Reagent Water³

3. Summary of Practice

3.1 A ground sample is weighed into a platinum crucible, and an appropriate amount of alkaline flux (Na₂CO₃ and Na₂B₄O₇) 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.

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.