Designation: C 1463 – 00

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

This standard is issued under the fixed designation C 1463; 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 C 169 for dissolution of waste containing glass in shielded facilities. Test Methods C 169 is not practical for use in such facilities and with radioactive materials.

1.7 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.

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:*

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[3]

C 1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste[3]

C 1285 Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)[3]

D 1193 Specifications for Reagent Water[4]

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, 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

[1] These practices are under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.05 on Analytical Test Methods.

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[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.

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