



Designation: **C1463—13 C1463 – 19**

# Standard Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis<sup>1</sup>

This standard is issued under the fixed designation C1463; 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 ( $\epsilon$ ) 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 C169 for dissolution of waste containing glass in shielded facilities. Test Methods C169 is not practical for use in such facilities and with radioactive materials.

1.7 The ICP-AES methods in Test Methods C1109 and C1111 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 C169.

1.8 Solutions from this practice may be suitable for analysis using ICP-MS after establishing laboratory performance criteria: criteria and verification that the criteria can be met. For example, Test Methods C1287 or C1637 may be used with additional sample preparation as necessary and appropriate matrix effect considerations.

1.9 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. Units in parentheses are for information only.

1.10 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Sections 10, 20, and 30.

1.11 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

C169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass

C859 Terminology Relating to Nuclear Materials

C1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy

C1111 Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectroscopy

<sup>1</sup> These practices are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.05 on Methods of Test. Current edition approved July 1, 2013/February 1, 2019. Published July 2013/February 2019. Originally approved in 2000. Last previous edition approved in 2007/2013 as C1463—00 (2007)-C1463 – 13. DOI: 10.1520/C1463-13.10.1520/C1463-19.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

- [C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste](#)  
[C1285 Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test \(PCT\)](#)  
[C1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry](#)  
[C1637 Test Method for the Determination of Impurities in Plutonium Metal: Acid Digestion and Inductively Coupled Plasma-Mass Spectroscopy \(ICP-MS\) Analysis](#)  
[D1193 Specification for Reagent Water](#)  
[E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves](#)

### 3. Terminology

- 3.1 For definitions of terms used in this Practice, refer to Terminology [C859](#).

### 4. Summary of Practice

- 4.1 The three practices for dissolving silicate matrix samples each require the sample to be dried and ground to a fine powder.
- 4.2 In the first practice, a mixture of sodium tetraborate ( $\text{Na}_2\text{B}_4\text{O}_7$ ) and sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) 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.
- 4.3 The second practice described in this standard involves fusion of the sample with potassium hydroxide (KOH) or sodium peroxide ( $\text{Na}_2\text{O}_2$ ) using an electric Bunsen burner, dissolving the fused sample in water and dilute HCl, and making to volume for analysis.
- 4.4 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 ( $\text{HNO}_3$ ) acids. Boric acid is added to the resulting solution to complex excess fluoride ions.
- 4.5 These three practices offer alternative dissolution methods for a total analysis of a glass sample for major, minor, and radionuclide components.

### 5. Reagents

- 5.1 *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.<sup>3</sup>
- 5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean at least Type II reagent water in conformance with Specification [D1193](#).

## PRACTICE 1—FUSION WITH SODIUM TETRABORATE AND SODIUM CARBONATE

### 6. Scope

6.1 This practice covers flux fusion sample decomposition and dissolution for the determination of  $\text{SiO}_2$  and 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.

### 7. Technical Precautions

- 7.1 This procedure is not useful for the determination of boron or sodium since these elements are contained in the flux material.
- 7.2 The user is cautioned that with analysis by ICP-AES, AAS, and ICP-MS, the high sodium concentrations from the flux may cause interferences.
- 7.3 Elements that form volatile species under these alkaline fusion conditions may be lost during the fusion process (that is, As and Sb).

### 8. Apparatus

- 8.1 *Platinum Crucibles*, 30 mL.
- 8.2 *Balance*, analytical type, precision to 0.1 mg.

<sup>3</sup> *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.

- 8.3 *Furnace*, with heating capacity to 1000°C.
- 8.4 *Crucible Tongs*, (cannot be made of iron, unless using platinum-clad tips).
- 8.5 *Polytetrafluoroethylene (PTFE) Beaker*, 125-mL capacity.
- 8.6 *Magnetic Stir Bar*, PTFE-coated (0.32 to 0.64 cm).
- 8.7 *Magnetic Stirrer*.
- 8.8 *Mortar and Pestle*, agate or alumina (or equivalent grinding apparatus).
- 8.9 *Sieves*, ~~100-mesh~~ 150  $\mu\text{m}$  (100 mesh), as described in Specification [E11](#).

## 9. Reagents and Materials

- 9.1 *Anhydrous Sodium Carbonate* ( $\text{Na}_2\text{CO}_3$ ).
- 9.2 *Anhydrous Sodium Tetraborate* ( $\text{Na}_2\text{B}_4\text{O}_7$ ).
- 9.3 *Sodium Nitrate* ( $\text{NaNO}_3$ ).
- 9.4 *Hydrochloric Acid* ( $\text{HCl}$ ), 50 % (v/v), made from concentrated hydrochloric acid (sp gr 1.19) and water.
- 9.5 *Nitric Acid* ( $\text{HNO}_3$ ), 50 % (v/v), made from concentrated nitric acid (sp gr 1.44) and water.

## 10. Hazards and Precautions

- 10.1 Follow established laboratory practices when conducting this procedure.
- 10.2 The operator should wear suitable protective gear when handling chemicals.
- 10.3 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.
- 10.4 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.
- 10.5 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.

## 11. Sample Preparation

- 11.1 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~~ 150  $\mu\text{m}$  (100-mesh) sieve using a clean mortar and pestle such as agate or ~~alumina~~ alumina.

## 12. Procedure

- 12.1 Weigh 50 to 250 mg of a powdered sample into a platinum crucible on an analytical balance to  $\pm 0.1$  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 entirely, and a smaller sample size may be necessary.

- 12.2 Add  $0.5 \pm 0.005$  g each of  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7$  to the crucible containing the sample.
- 12.3 Stir the sample/flux mixture in the crucible with a spatula until a mixture is obtained. Prepare a reagent blank.
- 12.4 For samples containing minor to major elements that do not oxidize readily (such as Pb, Fe, etc.), add 300 mg of sodium nitrate. If desired, a Pt lid can be placed on the crucible to reduce splattering. When adding nitrate, 50 % v/v  $\text{HNO}_3$  should be the diluting acid in order to reduce the attack on platinum in [12.6](#).
- 12.5 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.
- 12.6 Place a stir bar in each crucible and add 4 mL 50 % v/v  $\text{HCl}$ , and then dilute with  $\text{H}_2\text{O}$  to near the top of the crucible.

NOTE 2—In some cases, 50 % v/v  $\text{HNO}_3$  may be more appropriate than  $\text{HCl}$  (that is, samples for ICP-MS, high lead samples, or when sodium nitrate was added).

- 12.7 Place the crucible on the magnetic stirrer, and stir until the sample melt is dissolved completely (approximately 30 min). If undissolved material remains, the fusions described in Section [22](#) may need to be tried for cross correlation.
- 12.8 To a calibrated volumetric flask, typically 100, 250, 500, or 1000 mL, add enough 1:1  $\text{HCl}$  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.

12.9 The dilution volume is determined by the user of the practice and is dependent upon the desired analysis.

### 13. Precision and Bias

13.1 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.

13.2 See [Appendix X1](#) for examples of analytical data using solutions from this fusion.

## PRACTICE 2—FUSION WITH POTASSIUM HYDROXIDE OR SODIUM PEROXIDE

### 14. Scope

14.1 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.

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

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

### 15. Summary of Practice

15.1 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.

15.2 With appropriate sample preparation, the solution resulting from this procedure can be analyzed for trace metals by ICP-AES, ICP-MS, and AAS, and for radionuclides using applicable radiochemical methods.

### 16. Significance and Use

16.1 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.

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

16.3 This practice can be used for bulk analysis of glass samples for the product consistency test (PCT) as described in Test Methods [C1285](#) and for the analysis of monolithic radioactive waste glass used in the static leach test as described in Test Method [C1220](#).

16.4 This practice can be used to dissolve the glass reference and testing materials described in Refs [\(1\)](#) and [\(2\)](#).<sup>4</sup>

### 17. Interferences

17.1 Elements that form volatile species under these alkaline fusion conditions will be lost during the fusion process.

17.2 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.

17.3 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.

### 18. Apparatus

18.1 *Analytical Balance*, capable of weighing to  $\pm 0.1 \pm 0.1$  mg.

18.2 *Electric Bunsen Burner*, capable of heating to ~~1000°C~~ <sup>1000°C</sup><sup>5</sup> 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

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this practice.

<sup>5</sup> Electric Bunsen burners are available from most major laboratory supply houses.

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



FIG. 1 Electric Bunsen Burner Mounted on the Orbital Shaker

mounted on the orbital shaker with the above modifications for crucible mounting.

18.3 *Orbital Shaker*, including a holder fabricated to fasten the electric Bunsen burner on the platform (see Fig. 1).<sup>6</sup>

18.4 *Manual Adjustable Power Supply*, for controlling the temperature of the electric Bunsen burner.<sup>7</sup>

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

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

18.7 *Aluminum Oxide Crucible*, 55 mL capacity. Different shape and capacity may be used depending upon sample sizes taken.

18.8 *200 Mesh (74  $\mu\text{m}$ ) Sieve/Sieve, 75  $\mu\text{m}$  (200 Mesh)*, as described in Specification E11.

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

## 19. Reagents and Materials

19.1 *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.<sup>3</sup>

19.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean at least Type II reagent water conforming to Specification D1193.

19.3 *Potassium Hydroxide (KOH)*, pellet.

19.4 *Potassium Nitrate (KNO<sub>3</sub>)*, crystal.

19.5 *Sodium Peroxide (Na<sub>2</sub>O<sub>2</sub>)*, granular.

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

19.7 *Nitric Acid Solution (2 vol %)*—Add 20 mL of concentrated nitric acid (HNO<sub>3</sub>, sp gr 1.42) to 950 mL of water while stirring. Make to 1 L volume and store in a polyethylene bottle.

<sup>6</sup> Orbital shaker, Model 04732-00 available from Cole-Parmer Instrument Company, has been found to be suitable for this purpose.

<sup>7</sup> The Model 01575-26 power supply available from Cole-Parmer Instrument Company has been found to be suitable for this purpose.

19.8 *Oxalic Acid*, crystals.

## 20. Hazards and Precautions

20.1 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.

20.2 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.

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

20.4 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 %.

## 21. Sample Preparation

21.1 *Wet or Slurry Samples:*

21.1.1 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)~~ 75 μm (No. 200) sieve.

21.1.2 Weigh a portion (approximately 3 g) of the dried and ground sample described in 21.1.1 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:

$$I_F = (W_i - W_f)/(W_i) \quad (1)$$

where:

$W_i$  = initial sample weight, and

$W_f$  = sample weight after ignition.

21.2 *Dry Solid or Oxide Samples:*

21.2.1 Grind the dry solid or oxide sample to a particle size to pass a ~~No. 200 (74 μm)~~ 75 μm (No. 200) sieve.

21.2.2 Weigh a portion (approximately 3 g) of the ground sample described in 21.2.1 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 equation 21.1.2

NOTE 3—The loss on ignition for dry solid or oxide samples may be negligible.

## 22. Procedure

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

22.1.1 The choice of fusion methods described in 22.1 and 22.2 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.

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

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

22.1.4 Weigh an aliquot of the ground sample described in 21.1.1 or 21.2.1, which is equivalent to  $0.350 \pm 0.050$  g of ignited sample (21.1.2 or 21.2.2). Determine the amount of dried sample ( $W_s$ ) to be aliquoted by using the ignition factor from 21.1.2 as follows:

$$W_s = (0.350 \text{ g})/(1 - I_F) \quad (2)$$

22.1.5 Add  $1.600 \pm 0.200$  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.

22.1.6 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.

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

22.1.8 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 4—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.

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