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# Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)<sup>1</sup>

This standard is issued under the fixed designation C1285; 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 product consistency Test Methods A and B provide a measure of the chemical durability of homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, multiphase glass ceramic waste forms, or combinations thereof, hereafter collectively referred to as “glass waste forms” by measuring the concentrations of the chemical species released to a test solution under carefully controlled conditions.

1.1.1 Test Method A is a seven-day chemical durability test performed at  $90 \pm 2$  °C in a leachant of ASTM-Type I water. The test method is static and conducted in stainless steel vessels. The stainless steel vessels require a gasket to remain leak-tight (see [Note 1](#)) The stainless steel vessels are considered to be “closed system” tests. Test Method A can specifically be used to evaluate whether the chemical durability and elemental release characteristics of nuclear, hazardous, and mixed glass waste forms have been consistently controlled during production. This test method is applicable to radioactive and simulated glass waste forms as defined above.

NOTE 1—TFE-fluorocarbon gaskets, available commercially, are acceptable and chemically inert up to radiation doses of  $1 \times 10^5$  R of beta or gamma radiation which have been shown not to damage TFE-fluorocarbon. If higher radiation doses are anticipated, special gaskets fabricated from metals such as copper, gold, lead, or indium are recommended.

1.1.2 Test Method B is a durability test that allows testing at various test durations, test temperatures, particle size and masses of glass sample, leachant volumes, and leachant compositions. This test method is static and can be conducted in stainless steel or PFA TFE-fluorocarbon vessels. The stainless steel vessels are considered to be “closed system” while the PFA TFE-fluorocarbon vessels are considered to be “open system” tests. Test Method B can specifically be used to evaluate the relative chemical durability characteristics of

homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, or multiphase glass ceramic waste forms, or combinations thereof. This test method is applicable to radioactive (nuclear) and mixed, hazardous, and simulated glass waste forms as defined above. Test Method B cannot be used as a consistency test for production of high level radioactive glass waste forms.

1.2 These test methods must be performed in accordance with all quality assurance requirements for acceptance of the data.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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>
- C92 Test Methods for Sieve Analysis and Water Content of Refractory Materials
  - C162 Terminology of Glass and Glass Products
  - C169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass
  - C225 Test Methods for Resistance of Glass Containers to Chemical Attack
  - C371 Test Method for Wire-Cloth Sieve Analysis of Non-plastic Ceramic Powders

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.13 on Spent Fuel and High Level Waste.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

- [C429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture](#)
- [C693 Test Method for Density of Glass by Buoyancy](#)
- [C859 Terminology Relating to Nuclear Materials](#)
- [C1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy](#)
- [C1174 Guide for Evaluation of Long-Term Behavior of Materials Used in Engineered Barrier Systems \(EBS\) for Geological Disposal of High-Level Radioactive Waste](#)
- [C1463 Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis](#)
- [C1662 Practice for Measurement of the Glass Dissolution Rate Using the Single-Pass Flow-Through Test Method](#)
- [D859 Test Method for Silica in Water](#)
- [D1129 Terminology Relating to Water](#)
- [D1193 Specification for Reagent Water](#)
- [D1293 Test Methods for pH of Water](#)
- [D4327 Test Method for Anions in Water by Suppressed Ion Chromatography](#)
- [D5956 Guide for Sampling Strategies for Heterogeneous Wastes](#)
- [E7 Terminology Relating to Metallography](#)
- [E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)
- [E456 Terminology Relating to Quality and Statistics](#)
- [E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)
- [E1402 Guide for Sampling Design](#)

### 3. Terminology

#### 3.1 Definitions:

3.1.1 See Terminology [C859](#) for additional references not listed below.

3.1.2 *anneal, n*—to prevent or remove materials processing stresses in glass by controlled cooling from a suitable temperature (modified from Terminology [C162](#)).

3.1.3 *annealing, n—in glass leach tests*, a controlled cooling process for glass designed to reduce thermal residual stress to an acceptable level, and, in some cases, modify structure (Terminology [C859](#)).

3.1.4 *ASTM Type I water, n*—purified water with a maximum total matter content including soluble silica of 0.1 g/m<sup>3</sup>, a maximum electrical conductivity of 0.056 µmho/cm at 25 °C, a minimum electrical resistivity of 18 MΩ·cm at 25 °C (see Specification [D1193](#) and Terminology [D1129](#)).

3.1.5 *chemical durability, n—in leach tests*, the resistance of a material to alteration, dissolution and release of its constituents, under the specific conditions of the test (Terminology [C859](#)).

3.1.6 *closed system, n—in leach tests*, system utilizing a test container that is impervious to material transport (Terminology [C859](#)).

3.1.7 *consistently controlled, adj—in high level waste vitrification in the US*, that has been controlled in such a way that

its chemical durability is consistent, by comparison with a standard or a target, or by other experiments (proposed Terminology [C859](#)).

3.1.8 *devitrified glass, n*—an initially homogeneous or phase separated glass, or both, that has partially crystallized during cooling, heat treatment, or both (Terminology [C859](#)).

3.1.9 *glass, n*—an inorganic product of fusion that has cooled to a rigid condition without crystallizing (see Terminologies [C162](#) and [C859](#)).

3.1.10 *glass ceramic, n*—a solid material composed of both crystalline and glassy phases (Terminology [C859](#)).

3.1.11 *hazardous waste, n*—(1) *in waste management in a broad sense*, any substance or mixture of substances having properties capable of producing adverse effects on the health or safety of a human (see also *RCRA hazardous waste*); (2) *in waste management in the US*, any waste that is “listed” in 40CFR Parts 261.31 -261.33 or exhibits one or more of the characteristics identified in 40CFR Parts 261.20 -261.24, is a mixture of hazardous and non-hazardous waste, or is determined to be hazardous waste by the generator (proposed Terminology [C859](#)).

3.1.12 *hazardous waste glass, n*—a glass comprised of glass forming additives and hazardous waste.

3.1.13 *homogeneous glass, n*—a glass that is a single amorphous phase; a glass that is not separated into multiple amorphous phases (Terminology [C859](#)).

3.1.14 *leachant, n—in leach tests*, general term for the initial solution with which a solid is contacted and into which the solid dissolves or is leached (Terminology [C859](#)).

3.1.15 *leachate, n—in leach tests*, general term for the solution resulting from a test in which a solid is contacted by a solution and leaches or dissolves (Terminology [C859](#)).

3.1.16 *mixed waste, n—in the US*, waste containing radioactive, source special nuclear, or byproduct material regulated by the Atomic Energy Act (AEA) and hazardous components regulated by the Resource Conservation and Recovery Act (RCRA); the term “radioactive component” refers only to actual radionuclides dispersed or suspended in the waste substance (DOE Order 5400.3) (proposed Terminology [C859](#)).

3.1.17 *mixed waste glass, n—in nuclear waste management in the US*, a glass composed of glass-forming additives and mixed waste components (proposed Terminology [C859](#)).

3.1.18 *nuclear waste glass, n*—a glass composed of glass forming additives and radioactive waste (proposed Terminology [C859](#)).

3.1.19 *open system, n—in leach tests*, a system utilizing a test container through which material transport is possible, for example O<sub>2</sub> or CO<sub>2</sub> diffusion, or both (Terminology [C859](#)).

3.1.20 *phase separated glass, n*—a glass composed of more than one amorphous phase (Terminology [C859](#)).

3.1.21 *radioactive, adj*—of or exhibiting radioactivity (proposed Terminology [C859](#)).

3.1.22 *radioactivity, n*—spontaneous nuclear disintegration with emission of corpuscular or electromagnetic radiation, or both (consult Terminology D1129).

3.1.23 *sample blank, n*—a test in a cleaned test vessel that has been filled with the same amount of leachant as the tests with the waste form samples but containing no waste form sample that is conducted under the same conditions as tests with the waste form.

3.1.24 *sensitization, n*—in austenitic steels such as Types 304 and 316, the precipitation of chromium carbide at the grain boundaries in a temperature range of 400 to 900 °C (modified from Terminology E7).

3.1.24.1 *Discussion*—Sensitization constitutes the greatest single threat to their corrosion resistance (1).<sup>3</sup>

3.1.25 *set of samples, n*—samples tested simultaneously in the same oven.

3.1.26 *simulated waste glass, n*—a glass comprised of glass forming additives with simulants of, or actual chemical species, or both, in radioactive wastes or in mixed nuclear wastes, or both.

3.1.27 *standard, n*—to have the quality of a model, gage, pattern, or type (*Webster’s New Twentieth Century Dictionary, 1973*).

3.1.28 *standardize, v*—to make, cause, adjust, or adapt to fit a standard; to cause to conform to a given standard, for example, to make standard or uniform (*Webster’s New Twentieth Century Dictionary, 1973*).

3.1.29 *unsensitized austenitic steel, n*—stainless steel that is not sensitized (see **sensitization**).

3.1.30 *verify, v*—to determine or test the accuracy of, as by comparison, investigation, or reference, for example, to conduct experiments to verify a hypothesis (*The American Heritage Dictionary, 1973*).

3.1.31 *vitrification, n*—the process of fusing waste or simulated waste with glass making chemicals at elevated temperatures to form a waste glass or a simulated waste glass (proposed Terminology C859).

#### 4. Summary of Test Methods

4.1 Test Method A is the Product Consistency Test (PCT-A), which was developed specifically to measure the chemical durability of radioactive glass waste forms as defined in 1.1 during production (Table 1) (2). It can also be used to measure the chemical durability of hazardous, mixed, and various simulated glass waste forms as defined in 1.1. The test method is easily repeatable, can be performed remotely on highly radioactive samples and can yield results rapidly. The glass waste form does not need to be annealed prior to testing. In this test method, the glass waste form is crushed and sieved to isolate the size fraction between U.S. Standard ASTM –100 and +200 mesh sieves (0.150 to 0.075 mm) for use in the test, the particles are cleaned of adhering fines (see Note 3), and a weighed amount of sized and cleaned glass waste form that is

TABLE 1 Summary of Test Methods A and B

	Test Method A	Test Method B
Type of Waste Form	Radioactive Mixed Simulated, Hazardous	Radioactive Mixed Simulated, Hazardous
Usage	During production for rapid analysis and for waste compliance (3)	Scoping tests; Crystallization studies (see Note 3); Comparative waste form evaluation
Test Vessel	Unsensitized Type 304L stainless steel; vessels rated to >0.5 MPa (see Section 9)	Unsensitized Type 304L stainless steel or PFA TFE-fluorocarbon <sup>9</sup> vessels rated to >0.5 MPa (see Section 9)
Test Duration	7 days ± 2%	7 days ± 2 % or varying times
Leachant	ASTM Type I water	ASTM Type I water or other solutions
Condition	Static	Static
Minimum Sample Mass	≥ 1 g	≥ 1 g
Particle Size	U.S. Standard ASTM –100 to +200 mesh (0.150 to 0.075 mm)	U.S. Standard ASTM –100 to +200 mesh (0.150 to 0.075 mm) or other sizes which are <40 mesh (0.425 mm)
Leachant Volume	10 ± 0.1 cm <sup>3</sup> /g of sample mass	10 ± 0.1 cm <sup>3</sup> /g of sample mass or other volume/sample mass
Temperature	90 ± 2 °C	90 ± 2 °C or other temperatures provided that any observed changes in reaction mechanism are noted
Atmosphere	Air	Air or CO <sub>2</sub> free air (optional) (see Section 10)
Type of System	Closed to transport	Open to transport in PFA TFE-fluorocarbon; Closed to transport in stainless steel

greater than or equal to 1 g is placed in a Type 304L stainless steel vessel. An amount of ASTM Type I water equal to ten times the sample mass ( $m_{solid}$ ) (see Note 2) is added so that ( $V_{soln}/m_{solid}$ ) = 10 ± 0.1 cm<sup>3</sup>/g and the vessel is sealed. The vessel is placed in a constant temperature device at 90 ± 2 °C. The vessels must be placed in constant temperature devices so that there is ample convection around the vessels and even heat distribution (Fig. 1). After seven days ± 2 % (3.4 h), the vessel is removed from the constant temperature device and cooled to ambient temperature. The pH of an aliquot of the leachate is measured and temperature of the aliquot at the time of the pH measurement is also recorded. The remaining leachate is filtered and the filtrate sent for analysis. Tests with a reference glass are to be conducted in parallel with tests with the glass waste form to verify that the tests were conducted and analyzed properly. The test response provides a measure of the amounts of various glass components that are released to solution under carefully controlled conditions that can be expressed in terms of an average glass dissolution rate over the seven-day test

<sup>3</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.



FIG. 1 (a) Desensitized Type 304L Stainless Steel, 22 mL; PCT Vessel and Lid; White Polytetrafluoroethylene Gasket Which Seals Vessel and Lid When Assembly is Closed and Tightened; and Nickel-Plated Brass, Nut, and Screw Vessel Tightening Assembly



Vessels Should be Purchased with a Special Set of Plastic Wrenches (Not Shown) for Tightening.

FIG. 1 (b) 60 mL Polytetrafluoroethylene Vessel and Lid

interval. The most important elements to be analyzed in the leachate are those that are not sequestered in precipitates and not solubility limited; the solution concentrations of those elements are the best indicators of glass waste form durability. In the case of a multi-phase glass ceramic waste form, it may be important to analyze for elements that represent each significant phase. Extensive testing of any glass or glass ceramic waste form must be performed in order to identify elements that are representative of each phase (see Section 26).

NOTE 2—If waste forms of different densities are being compared then the leachate results from the test must be compared using the calculation in 25.3 which accounts for density differences in the SA/V term in the

denominator (see calculation in Appendix X1).

4.2 Test Method B is the Product Consistency Test (PCT-B), which was developed to measure the chemical durability of radioactive, mixed, or simulated glass waste forms (2). The test method is easily repeatable, can be performed remotely if necessary, and can yield results rapidly. The glass waste form does not need to be annealed prior to testing. In this test method the glass waste form is crushed and sieved to isolate the size fraction between U.S. Standard ASTM -100 and +200 mesh sieves (0.150 to 0.075 mm) for the use in the test or the size range of interest as long as the glass waste form particles are less than U.S. Standard ASTM 40 mesh (0.425 mm). The

particles are cleaned of adhering fines (see **Note 3**), and an amount of sized and cleaned glass waste form greater than or equal to 1 g is placed into either a Type 304 L stainless steel vessel or a PFA TFE-fluorocarbon vessel. An amount of ASTM Type I water equal to  $10 \pm 0.1 \text{ cm}^3/\text{g}$  of sample mass is added and the vessel is sealed. The use of other solution volume to sample mass ratios and other leachants are allowed. The vessel is placed in a constant temperature device at  $90 \pm 2 \text{ }^\circ\text{C}$ . Other test temperatures are permissible. It is desirable that inter-comparison of test responses be conducted at different temperatures to indicate whether the reaction mechanism changes over the temperature range investigated. The vessels must be placed in a constant temperature device so that there is ample convection around the vessels and even heat distribution (**Fig. 1**). After seven days or other test durations, the vessel is removed from the constant temperature device and cooled to ambient temperature. The pH is measured on an aliquot of the leachate and the temperature of the aliquot at the time of the pH measurement is also recorded. The remaining leachate is filtered and the filtrate sent for analysis. Tests with a reference glass are to be conducted in parallel with tests with the glass waste form to verify that the tests were conducted and analyzed properly. The test response provides a measure of the amounts of various glass components that are released to solution under carefully controlled conditions that can be expressed in terms of an average glass dissolution rate over the test interval. The most important elements to be analyzed in the leachate are those that are not sequestered in precipitates or solubility limited; those elements are good indicators of glass waste form durability. In the case of a multi-phase glass ceramic waste form, it may be important to analyze for elements from each significant phase present. Extensive testing of any glass or glass ceramic waste form must be performed in order to determine what these elements are (see **Section 26**). The results can be normalized to the glass surface area to solution volume ratio and glass composition to compare tests with different glasses reacted under different conditions. The results of tests conducted with the same test parameters but for different durations can be combined to describe the dissolution kinetics under the particular test conditions.

**NOTE 3**—Devitrified glasses, glass ceramics, and multiphase glass ceramic waste forms containing soluble secondary phases require special handling procedures (see **19.6.1** and **22.6.1**).

## 5. Significance and Use

5.1 These test methods provide data useful for evaluating the chemical durability (see **3.1.5**) of glass waste forms as measured by elemental release. Accordingly, it may be applicable throughout manufacturing, research, and development.

5.1.1 Test Method A can specifically be used to obtain data to evaluate whether the chemical durability of glass waste forms have been consistently controlled during production (see **Table 1**).

5.1.2 Test Method B can specifically be used to measure the chemical durability of glass waste forms under various test conditions, for example, varying test durations, test temperatures, sample surface area (SA)-to-leachant volume (V) ratios (see **Appendix X1**), and leachant types (see **Table 1**).

Data from this test may form part of the larger body of data that are necessary in the logical approach to long-term prediction of waste form behavior (see **Practice C1174**).

## 6. Apparatus

6.1 *Test Vessels for Test Method A*—The production test method requires the use of unsensitized Type 304L stainless steel leach vessels of >20 mL capacity designed with an internal pressure rating >0.5 MPa (see **Sections 10 and 11**).

6.1.1 The stainless steel vessels require a gasket to remain leak-tight. TFE-fluorocarbon gaskets, available commercially, are acceptable for test durations of less than 28 days since TFE-fluorocarbon is chemically inert and exposure to radiation doses up to  $1 \times 10^5$  rad of beta or gamma radiation have been shown not to damage TFE-fluorocarbon (**4**). If higher radiation doses are anticipated, degradation of the TFE-fluorocarbon gasket can compromise the seal and contaminate the leachate with  $\text{F}^-$  and HF (**5**). For high radiation doses, special gaskets fabricated from metals such as copper, gold, lead or indium are recommended.

6.2 *Test Vessels for Method B*—Test Method B allows for the use of either unsensitized Type 304L stainless steel or PFA TFE-fluorocarbon leach vessels of >20 mL capacity designed with pressure ratings >0.5 MPa (see **Section 10**).

6.2.1 The stainless steel vessels require a gasket material in order to remain leak-tight. If radioactive glass waste forms are tested in stainless steel vessels with TFE-fluorocarbon gaskets the same constraints that are noted in **6.1** for radioactive usage in Test Method A apply.

6.2.2 High radiation fields ( $>1 \times 10^5$  rad of beta or gamma radiation) will not generally be generated by hazardous, mixed or simulated nuclear waste glass forms. Commercially available PFA TFE-fluorocarbon vessels can be used in the absence of high radiation fields because PFA TFE-fluorocarbon is chemically inert when properly cleaned (**6**).<sup>4</sup>

**NOTE 4**—PFA TFE-fluorocarbon is perfluoroalkoxy TFE-fluorocarbon.

6.3 *Constant Temperature Devices*—Laboratory ovens or water baths capable of maintaining  $\pm 2.0 \text{ }^\circ\text{C}$  uniformity throughout the entire interior of the device, including the samples, at the test temperature are to be used for sample leaching and sample drying. These devices must be equipped with an over-temperature control.

6.4 *Conventional Oven*—Ovens, capable of maintaining  $\pm 10 \text{ }^\circ\text{C}$  can be used for vessel cleaning and sample drying.

6.5 *Temperature Measurement Device*—Resistance thermometers or thermocouples, or both, with a strip chart recorder or a data logger for periodic monitoring of the temperature of the convection oven during the test duration. The maximum period between temperature recordings should be 0.5 h.

<sup>4</sup> The sole source of supply of the PFA TFE-fluorocarbon manufactured without plasticizers or organic additives known to the committee at this time is Savillex Corp., 6133 Baker Rd., Minnetonka, MN 55345. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

6.6 *Balance(s)*—Any balance that will provide the following sensitivity: 0.25 % of the smallest masses to be measured, including the masses of the reagents, sample, leachant, leachate, leach vessel, and any required combinations.

6.7 *Weight Calibration Set*—A standard weight calibration set covering the range to include the smallest and largest weights to be measured. The weight calibration set should be traceable to the National Institute of Standards and Technology (NIST).

6.8 *Crushing Device*—Any mechanical or manual crushing device that will avoid iron (mild steel) contamination in the crushed waste form specimen (7). Crushing and grinding devices made of tungsten carbide, agate, sapphire, stainless steel, or dense alumina are acceptable.

6.9 *Sieves*—A nest of U.S. standard ASTM stainless steel or brass sieves. The nest shall include the cover and receptacle, and the largest and smallest mesh size sieves for the desired size range.

6.10 *Flasks*—Class A or calibrated volumetric laboratory ware.

6.11 *Pipettes*—Calibrated pipettes. Pipette tips that have either been cleaned, sterilized, or individually packaged to avoid contamination from handling.

6.12 *Syringes and Syringe Filters*—Sterilized, cleaned, or individually packaged syringes and mono or bidirectional 0.45 µm syringe filters (see Note 5).

NOTE 5—Commercially available cellulose acetate filters have been shown not to contaminate test solutions.

6.13 *Sample Vials*—Pretreated or individually packaged sample vials and caps.

6.14 *pH meter and probe*—pH meter and probe (for example, combination pH electrode) with an accuracy of ±0.1 pH units.

6.15 *Water Purification System*—Water purification system for producing ASTM-Type I water.

6.16 *Ultrasonic Cleaner*—Device to remove fines from crushed waste form materials.

6.17 *Analytic Equipment*—Equipment for measuring anion and cation content of the leachates and anion content of dilute solutions, for example, inductively coupled plasma-atomic emission spectrometry (see Test Methods C1109 or Ref 8, or both), atomic absorption spectrometry, ion chromatography (consult Test Method D4327 or Ref 8, or both), ion selective electrodes or colorimetric methods (consult Test Method D859).

## 7. Standards

7.1 *Reference Waste Form*—A reference waste form (see Refs 9-14 for example) of choice, similar in composition to the waste form being tested, must be tested in triplicate along with triplicate Method A or Method B tests with the waste forms to confirm the tests were conducted properly (15). The reference waste form composition should be traceable to NIST or an equivalent metrology institute. A single set of triplicate tests

with the reference waste form may be conducted in parallel with triplicate tests of several waste forms.

7.2 *Multi-element Solution Standard*—A reference solution of choice, similar in composition to the leachate being tested, is to be submitted in triplicate along with each batch of leachates for multi-element analysis. The reference solution standard should be traceable to NIST, or an equivalent metrology institute, and have a certified shelf life.

7.3 *pH Buffers*—commercial pH buffers or pH buffers made to the specifications given in Test Method D1293 that bracket the measured pH range of the leachant and leachate. All commercial buffer solutions should be traceable to NIST, or an equivalent metrology institute, and have a certified shelf life. Keep all the reference buffer solutions well sealed and replace at the expiration of shelf life, or sooner if a visible change is observed (see Test Method D1293).

7.4 *Analytic Standard Solutions*—The reference solutions should be traceable to NIST, or an equivalent metrology institute. All standard solutions must have a certified shelf life.

## 8. Reagents and Standards

8.1 *ACS Reagent Grade Acids*—Reagent grade nitric acid (HNO<sub>3</sub>) and hydrofluoric acid (HF) for cleaning leach vessels.<sup>5</sup>

8.2 *ACS High Purity Acid*—Ultra high purity concentrated nitric acid (HNO<sub>3</sub>) for acidification of leachates.<sup>5</sup>

8.3 *Reagent Grade NaOH*—Reagent grade NaOH for cleaning of new PFA TFE-fluorocarbon vessels.

8.4 *Solvents*—Absolute ethanol (99 % pure) or another non polar solvent like cyclohexane or reagent grade acetone.

8.5 *ASTM Type I Water*—Type I water shall have a minimal electrical resistivity of 18 MΩ·cm at 25 °C (see Specification D1193).

8.5.1 The source water shall be purified, then passed through a deionizer cartridge packed with a mixed bed of nuclear-grade resin (see Note 6), then through a cellulose ester membrane having openings not exceeding 0.45 µm (see Note 7).

NOTE 6—A nuclear-grade resin mixture of the strong acid cation exchanger in the hydrogen form and the strong base anion exchanger in the hydroxide form with a one-to-one cation to anion equivalence ratio is suitable.

NOTE 7—An in-line filter has been found to be satisfactory.

8.5.2 Pass the purified water through an in-line conductivity cell to verify its purity. Alternatively, the water can be measured for all anions and cations to verify that there is less than a total dissolved solid content including soluble silica of 0.1 g/m<sup>3</sup> (see Specification D1193 and Terminology D1129).

8.6 *Other Leachants*—Test Method B allows for the use of other leachants such as simulated or real groundwaters, brine, seawater, pH buffers, and others. The simulated solutions should be made from ACS reagent grade chemicals.<sup>5</sup> All

<sup>5</sup> Available from American Chemical Society, 1155 16th St., NW, Washington, DC 20036.

leachants should be chemically analyzed to verify their composition before durability testing begins. All leachants should be used within their specified shelf life.

## 9. Hazards

9.1 All appropriate precautions for operation of pressurized equipment must be taken. To ensure safe operation, the test vessels should be designed to withstand the vapor pressure of water at the test temperature with an appropriate safety factor. The thermal expansion of water must be taken into account when filling the leach containers. Specifically, between 4 and 100 °C, water expands by 4 volume %. Overfilling, for example, filling a 60 mL vessel to 58 mL, may lead to pressures inside the container that exceed the design limits and could lead to the failure of one or more parts of the vessel.

## 10. Choice of Test Vessel

10.1 *Stainless Steel Vessels*—Unsensitized Type 304L stainless steel vessels must be used in Test Method A and may be used in Test Method B. The user should ensure that the vessels are free from chloride as radiolysis of Cl containing solutions can generate reducing species such as Cl<sub>2</sub> and various oxychlorides that can lower the redox (reduction/oxidation) potential of the leachate and give anomalous leach rates or attack the leach vessel, or both (16). The user is also cautioned that radionuclides such as americium, plutonium, and other redox sensitive species are known to plate-out on steel so that an acid strip of the interior of the vessel may be needed to account for all the radionuclides released (see 14.1.2).

10.1.1 Steel vessels represent “closed system” applications where the influx of CO<sub>2</sub> or O<sub>2</sub> into the leachate is not desired.

10.1.2 It is recommended that 22 mL steel vessels<sup>6</sup> be used for the radioactive production application in Test Method A (see Fig. 1a). This allows for the use of up to 18 cm<sup>3</sup> water to minimize the amount of radioactive sample being handled.

10.2 *PFA TFE-fluorocarbon Vessels*—PFA TFE-fluorocarbon vessels may be used in Test Method B (see Fig. 1b). PFA TFE-fluorocarbon vessels can be used for Test Method B for short-term chemical durability testing with mixed or simulated nuclear glass waste forms. The use of PFA TFE-fluorocarbon vessels is acceptable for test durations up to 28 days. Longer test durations are acceptable only if it can be demonstrated that the vessel interactions do not affect the glass waste form reactivity, including leaching of fluoride ions from the vessel and ingress of CO<sub>2</sub> from the atmosphere. The user should ensure that new PFA TFE-fluorocarbon vessels are free of leachable fluoride which is present as a free surface fluoride residue from vessel fabrication (see Section 16).

10.2.1 PFA TFE-fluorocarbon vessels are for “open system” applications where the influx of CO<sub>2</sub> or O<sub>2</sub> into the leachant is either desirable or not of concern. The user is cautioned that the leachate concentrations and leachate pH in PFA TFE-

fluorocarbon and steel vessels may be different due to equilibration with CO<sub>2</sub> and O<sub>2</sub>, that is, the differences in “open” and “closed” system conditions. The user is cautioned that the leachate concentrations and leachate pH values may be significantly different in tests conducted in PFA TFE-fluorocarbon and steel vessels due to higher dissolved concentrations of CO<sub>2</sub> and O<sub>2</sub> in tests performed in PFA TFE-fluorocarbon vessels, which provide “open system” conditions. (11, 12, 17-23)

10.2.2 PFA TFE-fluorocarbon vessels cannot be used in Test Method A and it is recommended that PFA TFE-fluorocarbon vessels not be used in Test Method B with radioactive glass waste forms.<sup>4</sup> The use of PFA TFE-fluorocarbon vessels under radiation doses >10<sup>5</sup> roentgen causes degradation of the PFA TFE-fluorocarbon, and subsequent release of F<sup>-</sup> and HF into the test solution. The presence of HF in the solution may change the rate of degradation of the glass waste form due to the acidification and F<sup>-</sup> ions that attack the glass waste form (4, 5) and lead to a spurious and erroneous test result.

## 11. Identification of Vessels and Vessel Cleaning History

11.1 *Identification of Vessels*—A unique identifying number should be permanently marked on each vessel. The same number should be permanently marked on the companion lid.

11.2 *Identification of Vessel Cleaning History*—Each batch of cleaned vessels will be labeled with a unique batch number. A log book of the vessel number and date the cleaning is completed shall be kept. The date can be used as the batch number identifier if only one batch has been cleaned on that date.

11.2.1 Alternatively, a separate batch number can be assigned and recorded in the log book. In this manner, any inconsistent test responses might be traced to insufficient or improper cleaning of a batch of vessels or to a problem vessel.

11.2.1.1 The batch number of the vessel used for each sample and blank while conducting PCT Test Method A or B will be entered on a model data sheet like the one in Appendix X2. These data will be maintained in a laboratory notebook for control purposes.

## 12. Cleaning of New Stainless Steel Vessels for PCT Test Methods A and B

12.1 New Type 304L stainless steel vessels shall be cleaned by the following procedure:

12.1.1 Remove any gaskets before cleaning new stainless steel vessel lids. Degrease the vessels and lids with acetone. Check the integrity of the gasket and discard if visibly damaged. Clean new undamaged TFE-fluorocarbon gaskets according to Section 13. Clean new undamaged metallic gaskets according to 12.1.2 – 12.1.5.

12.1.2 Clean the vessels and lids ultrasonically in either 99 % ethanol or absolute ethanol or another non polar solvent like cyclohexane for approximately 5 min.

12.1.3 Rinse the vessels and lids three times with ASTM Type I water.

12.1.4 Submerge the vessels and lids in 0.16 M HNO<sub>3</sub> (1 weight % HNO<sub>3</sub>) and heat to 90 ± 10 °C for a minimum of 1 h.

<sup>6</sup> The sole source of supply of the apparatus known to the known to the committee at this time is Parr Instrument Co., 211 53rd St., Moline, IL 61265. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

12.1.5 Rinse the vessels three times with ambient temperature ASTM Type I water.

12.1.6 Submerge the vessels and lids in fresh ASTM Type I water for 1 h at  $90 \pm 10$  °C.

12.1.7 Rinse with fresh ASTM Type I water at ambient temperature.

12.1.8 Carefully place a cleaned gasket (see 13.1) in the lid. Fill the vessel 80 to 90 % full of ASTM Type I water. Close the lid and leave in a  $90 \pm 10$  °C oven for a minimum of 16 h.

12.1.9 Remove the vessels from the oven, cool to ambient temperature, take a cooled aliquot of the water and measure the pH (see Test Methods D1293).

12.1.10 If the pH is not in the range 5.0 to 7.0, repeat 12.1.6 – 12.1.9.

12.1.11 If the 5.0 to 7.0 pH range cannot be achieved by three repetitions of 12.1.6 – 12.1.9, then repeat the cleaning method starting at 12.1.4.

12.1.12 Dry vessels and lids at  $90 \pm 10$  °C for a minimum of 16 h and then cool them. If the vessels are not used immediately, close the vessels and store in a clean environment until needed.

### 13. Cleaning of New TFE-fluorocarbon Gaskets for Stainless Steel Vessels for PCT Test Methods A and B

13.1 New gaskets for stainless steel vessels should be cleaned by the following method:

13.1.1 Remove visible grease or dirt from acceptable gaskets using a clean lint free cloth and absolute ethanol. Recheck the integrity of the gasket and discard if damaged.

13.1.2 Handle the gaskets only with clean tongs or cotton gloves.

13.1.3 Clean each gasket ultrasonically in 99 % or absolute ethanol or another non polar solvent like cyclohexane for approximately 10 min.

13.1.4 Rinse each gasket in ASTM Type I water at ambient temperature for approximately 3 min.

13.1.5 Bake each gasket in an oven at  $200 \pm 10$  °C for a minimum of 4 h.

13.1.6 Immerse each cooled gasket in fresh ASTM Type I water in a boiling water bath for a minimum of 2 h.

13.1.7 Dry gaskets at  $90 \pm 10$  °C for a minimum of 16 h, and store in a clean environment until needed.

### 14. Cleaning of Used Stainless Steel Vessels for PCT Test Method A

14.1 When stainless steel vessels are reused subsequent to their use with radioactive materials, residual contamination may be present. The vessels shall be cleaned before reuse by cleaning with dilute HNO<sub>3</sub> and ASTM Type I water until the level of the radioactive element(s) of interest is (are) below the detectable level using the analytical method employed for concentration measurement of the leachate. Stainless steel vessels are also checked for silicon contamination before reuse. Used stainless steel containers for radioactive service (PCT Method A) shall be cleaned according to the following method:

14.1.1 Remove any remaining glass waste form sample from previous testing by rinsing the vessel and lid with ASTM Type I water. Fill the vessel 80 to 90 % full with 0.16 M HNO<sub>3</sub> (1 weight % HNO<sub>3</sub>). Reseal the vessel and place in  $90 \pm 10$  °C

oven for a minimum of 16 h to dissolve (acid strip) radionuclides adhering to the interior of the vessel.

14.1.2 Check the acid strip solution for radioactivity. Repeat 14.1.1 until the radioactivity of the acid strip solution is less than three times background.

14.1.3 If possible, remove the gasket and discard. Gaskets that have been exposed to HNO<sub>3</sub> during cleaning or acid stripping of the vessel may be compromised because small amounts of HNO<sub>3</sub> may be trapped between the gasket and the lid. Rinse vessels and lids thoroughly with deionized water and then with ASTM Type I water at ambient temperature. Extreme caution should be exercised so that the inside of the vessel is not contaminated with radioactivity that may have contacted the outside of the vessel.

14.1.4 Fill the vessel 80 to 90 % full of fresh ASTM Type I water. If necessary, put a new gasket in the lid. Close the vessel and leave in a  $90 \pm 10$  °C oven for a minimum of 24 h.

14.1.5 Remove vessels from oven, take one aliquot of water from each vessel and measure the pH (see Test Methods D1293). Take additional aliquots of water from each vessel and measure the radioactivity and the silicon content of the solution.

14.1.6 If the pH is not in the range 5.0 to 7.0, or the measured radioactivity is greater than three times background, or silicon is detected at  $>0.1$  g/m<sup>3</sup> in the solution by methods such as ICP or Test Method D859, repeat 14.1.3 – 14.1.5.

14.1.7 If the pH is not in the 5.0 to 7.0 range, or the measured radioactivity is greater than three times background, or the  $>0.1$  g/m<sup>3</sup> criteria cannot be achieved by three repetitions of 14.1.3 – 14.1.6, or a combination thereof, then repeat the cleaning method starting at 14.1.1.

14.1.8 Dry vessels, lids, and gaskets at  $90 \pm 10$  °C for a minimum of 16 h and store in a clean environment until needed.

### 15. Cleaning of Used Stainless Steel Vessels for PCT Test Method B

15.1 When stainless steel vessels are reused subsequent to their use with radioactive mixed waste specimens, residual contamination may be present. The vessels shall be cleaned before reuse by cleaning with HNO<sub>3</sub> and ASTM Type I water until the level of the radioactive element(s) of interest is (are) below the detectable level using the analytical method employed for concentration measurement of the leachate. Stainless steel vessels are also checked for silicon contamination before reuse. Used stainless steel containers for PCT Test Method B shall be cleaned according to the following method:

15.1.1 For stainless steel vessels that have been used for mixed waste glass form testing follow 14.1.1 and 14.1.2. For vessels used for non-radioactive glass waste form testing remove any remaining glass waste form from previous testing by rinsing the vessel and lid with ASTM Type I water.

15.1.2 Remove the gasket from the lid and discard. Gaskets that have been exposed to HNO<sub>3</sub> during cleaning or acid stripping of the vessel may be compromised because small amounts of HNO<sub>3</sub> may be trapped between the gasket and the lid.



15.1.3 Soak the vessels and lids in 0.16 M HNO<sub>3</sub> (1 weight % HNO<sub>3</sub>) at 90 ± 10 °C for 1 h.

15.1.4 Rinse vessels and lids thoroughly with ASTM Type I water at ambient temperature.

15.1.5 Heat vessels and lids in ASTM Type I water at 90 ± 10 °C for a minimum of 1 h.

15.1.6 Put a new gasket in the lid. Fill the vessel 80 to 90 % full of fresh ASTM Type I water. Close the lid and leave in a 90 ± 10 °C oven for a minimum of 24 h.

15.1.7 Remove vessels from oven, take one aliquot of water from each vessel and measure the pH (see Test Methods **D1293**). Take additional aliquots of water from each vessel and measure the silicon content of the solution.

15.1.8 If the pH of the aliquot is not in the range 5.0 to 7.0 or silicon is detected at >0.1 g/m<sup>3</sup> in the solution by methods such as ICP or Test Method **D859**, repeat steps **15.1.4 – 15.1.7**.

15.1.9 If the 5.0 to 7.0 pH range or the >0.1 g/m<sup>3</sup> silicon criteria cannot be achieved after three repetitions of **15.1.4 – 15.1.7**, then repeat the cleaning and testing method starting at **15.1.2**.

15.1.10 Dry vessels, lids, and gaskets at 90 ± 10 °C for a minimum of 16 h and store in a clean environment until needed.

## 16. Cleaning of New PFA TFE-fluorocarbon Vessels for PCT Test Method B

16.1 New PFA TFE-fluorocarbon vessels are cleaned before use with NaOH and ASTM Type I water to remove any free fluoride from the interior surfaces (6). New TFE-fluorocarbon leach containers shall be cleaned according to the following method:

16.1.1 Rinse PFA TFE-fluorocarbon vessels and lids with fresh ASTM Type I water at ambient temperature.

16.1.2 Fill vessels at least 90 % full with 5 weight % NaOH solution.

16.1.3 Tighten lids and place vessels in a preheated 110 ± 10 °C oven for at least seven days.

16.1.4 After 12 to 24 h remove the vessels from the oven long enough to retighten the lids.

16.1.5 Remove the vessels from the oven after the seven days and allow cooling to ambient temperature.

16.1.6 Open the vessels carefully and dispose of the NaOH solution.

16.1.7 Rinse the vessel and lid twice with fresh ASTM Type I water at ambient temperature.

16.1.8 Place the vessels and lids in fresh boiling ASTM Type I water for a minimum of 1 h.

16.1.9 Repeat **16.1.7** and **16.1.8**.

16.1.10 Fill the PFA TFE-fluorocarbon vessels at least 90 % full with fresh ASTM Type I water at ambient temperature. Close the vessels and leave in a 90 ± 10 °C oven for a minimum of 16 h.

16.1.11 Remove vessels from oven. Allow vessels to cool to ambient temperature. Take an aliquot of water from each vessel and measure the pH (consult Test Methods **D1293**).

16.1.12 If the pH is in the 5.0 to 7.0 pH range, check the F<sup>-</sup> concentration of the water in each vessel by measuring the F<sup>-</sup> concentration of another aliquot of the water from each vessel.

16.1.13 If the pH is <5.0 or the F<sup>-</sup> content is >0.5 µg/mL, repeat steps **16.1.1 – 16.1.12**.

16.1.14 If the pH is above 7.0 repeat steps **16.1.7 – 16.1.12**.

16.1.15 Dry vessels and lids at 90 ± 10 °C for a minimum of 16 h, and store in a clean environment until needed.

## 17. Cleaning of Used PFA TFE-fluorocarbon Vessels for PCT Test Method B

17.1 When PFA TFE-fluorocarbon vessels are reused, residual contamination from the glass waste forms tested may be present. The vessels shall be cleaned before reuse by cleaning with HNO<sub>3</sub> and ASTM Type I water. As a precaution fluoride contamination should continue to be checked for the first five uses of a given PFA TFE-fluorocarbon vessel. Used PFA TFE-fluorocarbon containers shall be cleaned according to the following method:

17.1.1 Remove any glass from previous waste form testing from the vessels by rinsing both the vessels and lid with ASTM Type I water.

17.1.2 Soak vessels and lids in 0.16 M HNO<sub>3</sub> (1 weight % HNO<sub>3</sub>) at 90 ± 10 °C for approximately 1 h.

17.1.3 Rinse vessels and lids thoroughly with fresh ASTM Type I water at ambient temperature.

17.1.4 Put vessels and lids in fresh ASTM Type I water at 90 ± 10 °C. Remove after approximately 1 h.

17.1.5 Fill each vessel 80 to 90 % full of fresh ASTM Type I water at ambient temperature. Close the lid and leave in a 90 ± 10 °C oven for a minimum of 16 h.

17.1.6 Remove vessels from oven, take an aliquot of water from each vessel and measure the pH (see Test Methods **D1293**).

17.1.7 If the pH is in the 5.0 to 7.0 pH range, check the F<sup>-</sup> concentration by measuring the F<sup>-</sup> content of another aliquot of the water. If a given vessel has been reused a minimum of five times and the vessel cleaning history indicates that the F<sup>-</sup> concentration has consistently been <0.5 µg/mL when the pH measurement is between 5.0 to 7.0, then the measurement of the solution pH is considered sufficient evidence that the solution is free of F<sup>-</sup> contamination.

17.1.8 If the pH is <5.0 or the F<sup>-</sup> content is >0.5 µg/mL, repeat **17.1.4** to **17.1.7**.

17.1.9 If the 5.0 to 7.0 pH range or the F<sup>-</sup> content cannot be achieved by three repetitions of **17.1.4** to **17.1.7**, then repeat the cleaning and testing method starting at **17.1.2**.

17.1.10 Dry vessels and lids at 90 ± 10 °C for a minimum of 16 h, and store in a clean environment until needed.

## 18. Calibration

18.1 *Calibrations*—Initially calibrate all instruments used in this test. Verify the calibrations during use of the instrument to indicate possible errors due to instrumental drift.

18.2 *Calibration and Standardization Schedule:*

18.2.1 *Temperature Measurement Devices*—Calibrate at least annually with standards traceable to NIST or an ice/boiling water bath.

18.2.2 *Balance*—Standardize before each use and after completion of all weighings with NIST standard masses. Have the balance calibrated on an annual basis.

18.2.3 *pH meter*—Standardize the pH meter before each use and after completion of all samples with commercial buffer solutions that bracket the solution pH values being measured. Standardize the pH meter at the same temperature as the leachate solutions being measured. If only an occasional pH determination is made, standardize the assembly each time it is used. In a long series of measurements, supplemental interim checks at regular intervals are recommended. In as much as commercially available pH assemblies exhibit different degrees of measurement stability, conduct these checks at intervals of 30 min, unless it is ascertained that less frequent checking is satisfactory to ensure the performance described in Test Methods **D1293**.

18.2.4 *Water Purification System*—Calibrate at least annually following the manufacturer's instructions. Standardize before every use with the 10 MΩ·cm at 25 °C resistivity calibration cell on the water purification system (see Test Methods **D1129** and **D1193**).

## PRODUCT CONSISTENCY TEST (PCT)—TEST METHOD A

### 19. Sample Preparation for PCT Test Method A

19.1 *Sample Handling*—All glass waste forms must be handled with clean equipment and stored in clean containers. For highly radioactive glass waste forms, when operations must be performed in a hot cell with manipulators, as much care as possible must be taken to avoid contamination during these sample preparation steps.

19.2 *Choice of Appropriate Sample*—Samples of glass waste forms may either be fabricated individually or taken from larger samples (see Terminologies **E456** and **E1402**). The user is cautioned that a representative sample should include the same proportional amounts of vitrified and devitrified (crystalline) phases found in the bulk sample to be meaningful. This can be achieved by taking sub-samples from more than one location and not taking sample surfaces that have air cooled, have a visibly different appearance, or have cooled against a metal container (consult **Appendix X1** of Guide **D5956**). The glass waste form does not have to be annealed. Flush the sample surface with ASTM Type I water to remove potential surface contamination and dry before crushing.

19.3 *Choice of Sample Mass*—Sample masses must be  $\geq 1$  g. The reference ratio of leachant volume to sample mass ( $V_{\text{soln}}/m_{\text{solid}}$ ) is  $10 \pm 0.1$  cm<sup>3</sup>/g. The volume of leachant is established by the mass of sample used in the test and both are constrained by the volume of the test vessel chosen. For example, 1.5 g of sample can be tested using between 14.25 and 15.75 cm<sup>3</sup> of leachant contained in a 22 mL steel vessel.

19.4 *Number of Sample Replicates*—A minimum of three replicate samples and three tests shall be used to provide a measure of the experimental variability.

19.5 *Crushing and Sieving Glass Waste Forms*—If the glass waste form is redox sensitive, the user is cautioned that grinding the waste form in advance of the test may cause the waste form to oxidize during exposure to air and may alter the leachate results. Redox sensitive glass waste forms shall be

used within two days of grinding (see Test Methods **C169** and Ref **24**). If the sample has dimensions larger than 2 cm, wrap the sample in a clean plastic bag and break it into smaller fragments with a hammer. It may be necessary to use steel crushing devices. Mild steel should be avoided due to the known interactions of mild steel and glass waste forms in solution (**7**). Crushing devices made of Types 304L and 316L stainless steel should be used to minimize these effects. It is recommended that the amount of the material prepared shall weigh at least twice the required weight of the sample needed to perform the PCT in triplicate in order to have enough sized sample to test.

19.5.1 Transfer waste form fragments into a clean manual or mechanical grinder. Clean the grinder prior to crushing a different waste form. Do not use mechanical grinders with steel blades unless they are known to be made of Types 304L or 316L stainless steel because of the known interactions of mild steels and glass waste forms in solution (**7**). If a laboratory size grinding mill is used, ensure that the blade is tungsten carbide and not mild steel. Because of the brittle nature of the tungsten carbide blades, glass waste form samples should be less than 1.0 cm before using the grinding mill. The sample basket of laboratory grinding mills should be made of stainless steel. If the sample basket appears dull due to erosion of the stainless steel, replace the sample basket. Use of mild steel baskets is not allowed (see **6.8**).

19.5.2 Clean the brass or stainless steel sieves, catch pan and lid before and after every use (see **Note 8**).

**NOTE 8**—It is recommended that brass or stainless steel sieves should be cleaned by flushing them with deionized water from all directions. Dry immediately with high pressure air or in an oven at 90 to 110 °C. Do not use solvents or high temperatures to clean brass sieves, as the brass mesh has a protective film to inhibit corrosion.

19.5.3 Visually inspect the sieves for holes or tears before every use. Do not use a torn or deformed sieve. Transfer crushed glass waste form to a clean nest of sieves. No more than 100 g of crushed waste form should be placed on an 8 in. diameter sieve (see Test Methods **C225** and **C371**; **Note 9**). Scaling the amount of waste form to the exposed surface area of the sieve indicates that no more than 50 g of crushed waste form should be placed on a 5 in. diameter sieve and no more than 25 g should be placed on a 3 in. diameter sieve. The correct amount of crushed waste form should be placed on a 100 mesh (0.150 mm) sieve. A 200 mesh (0.075 mm) sieve should be under the 100 mesh sieve with a catch pan below.

**NOTE 9**—The variability of the sample surface area to the solution volume has the largest effect on the precision of the test (**25, 26**).

19.5.4 Place the lid on the nest of sieves and sieve mechanically or manually as follows:

19.5.4.1 *Mechanical Sieving*—The mechanical shaking device<sup>7</sup> shall produce a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample

<sup>7</sup> The sole source of supply of the Ro-Tap Testing Sieve Shaker known to the committee at this time is W.S. Tyler Co., Mentor, OH 44060. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

moving continuously over the surface of the sieve (see Test Method C371). The mechanical sieving should continue for a minimum of 15 min (see Test Method C429).

19.5.4.2 *Hand Sieving*—Alternately, tap and rotate the nested sieves while holding them in a slightly inclined position so that the test sample will be well distributed over the sieve (see Test Methods C92). Sieve for several minutes.

19.5.5 Remove the 100 mesh sieve containing +100 mesh waste form fraction. Then remove the 200 mesh sieve containing the –100 to +200 mesh fraction.

19.5.6 Tap the 200 mesh sieve forcefully over contrasting colored paper. For example, if the waste form is light colored tap the sieve on dark paper. For a dark colored waste form, for example, black nuclear waste glass, tap the sieve on white paper. Tap the sieve in an inclined position on one side; repeat several times tapping the inclined sieve on opposite sides each time. Continue the tapping until no visible glass passes through the sieve onto the paper after 1 min of continuous tapping (see Test Methods C92).

19.5.7 Transfer the –100 to +200 mesh fraction of the sieved glass waste form into a clean container labeled with the sample and size fraction identification. The date and name of the person preparing the sample should also appear on the container.

19.5.8 If additional material is needed, crush the +100 mesh size waste form fragments again or begin again at step 19.5.1. After new waste form fragments have been prepared, repeat 19.5.3 – 19.5.7.

19.5.9 *Wet Sieving*—If the sample is friable or could give off radioactive dust it may be best to wet sieve with water, absolute ethanol, or another non polar solvent like cyclohexane by

forcefully squirting the sample with the liquid while the sample is on the sieve and over a catch vessel. Water can be used if there are no soluble phases in the sample. Either ethanol or another non polar solvent like cyclohexane should be used if soluble phases are known to be present or suspected to be present in the sample. Typically wet sieving is conducted for analysis of particles finer than about 200 mesh (0.075 mm), where powder surfaces develop enough of a surface charge and show an enhanced tendency to agglomerate or to minimize dusting from radioactive samples (27).

19.5.10 Enter sample identification, date, and name of the person preparing the sample on a sample log sheet such as that given in Appendix X2.

19.6 *Washing the –100 to +200 Mesh Glass Waste Form:*

19.6.1 Washing of the –100 to +200 mesh glass waste form to remove adhering fines is required for homogeneous glass waste forms containing no secondary phases (see Fig. 2). However, devitrified, phase separated, multiphase glass ceramic waste forms, or combinations thereof, may contain soluble secondary phases that might be substantially removed from the sample during the washing process. This possibility must be addressed when the test is used to determine the effect of phase separated crystalline or amorphous phases on the test response. Separated phases (crystalline or amorphous) that are present should be identified by X-ray Diffraction or scanning electron microscopy, or both, and either their potential to dissolve faster than the other phases in the bulk waste form be taken into account when analyzing the test results or the wash solutions analyzed directly. If preferential dissolution of a separated phase is likely in the washing step, then 19.6.3 –



NOTE 1—The polytetrafluoroethylene vessels have white crushed glass in the bottom of the containers and that special racks are used to allow the forced air to circulate around the leach vessels to maintain the vessel and its contents at constant temperature.

FIG. 2 Forced-air Circulation Blue M Oven with Numerous 304L Stainless Steel and Two Polytetrafluoroethylene Vessels