Standard Test Methods for
Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)[1]

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 (ε) 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 ± 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.[2] 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.

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. No other units of measurement are included in this 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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
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

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[1] 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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[2] TFE-fluorocarbon gaskets, available commercially, are acceptable and chemically inert up to radiation doses of 1×10^5 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.

[3] 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.

C1174 Practice for Prediction of the Long-Term Behavior of Materials, Including Waste Forms, Used in Engineered Barrier Systems (EBS) for Geological Disposal of High-Level Radioactive Waste
C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of 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:*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^3, 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 40CFRParts 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_2 or CO_2 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 400–900°C (modified from Terminology E7).

3.1.24.1 *Discussion*—This constitutes the greatest single threat to their corrosion resistance (**1**).[4]

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 (proposedTerminology 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 of U.S. Standard ASTM – 100 to + 200 mesh (0.149–0.074 mm) for use in the test, the particles are cleaned of adhering fines (see Note 1), and a weighed amount of sized and cleaned glass waste form that is 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})[5] is added so that (V_{soln}/m_{solid}) = 10 ± 0.5 mL/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 ±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

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 1); 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[9] 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 (0.149 to 0.074 mm)	U.S. Standard ASTM – 100 to + 200 mesh (0.149 to 0.074 mm) or other sizes which are <40 mesh (0.420 mm)
Leachant Volume	10 ± 0.5 cm^3/gram of sample mass	10 ± 0.5 cm^3/gram 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$_2$ 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

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[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[5] 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 which adjusts the leachate results for sample density (see calculation in Appendix X1).

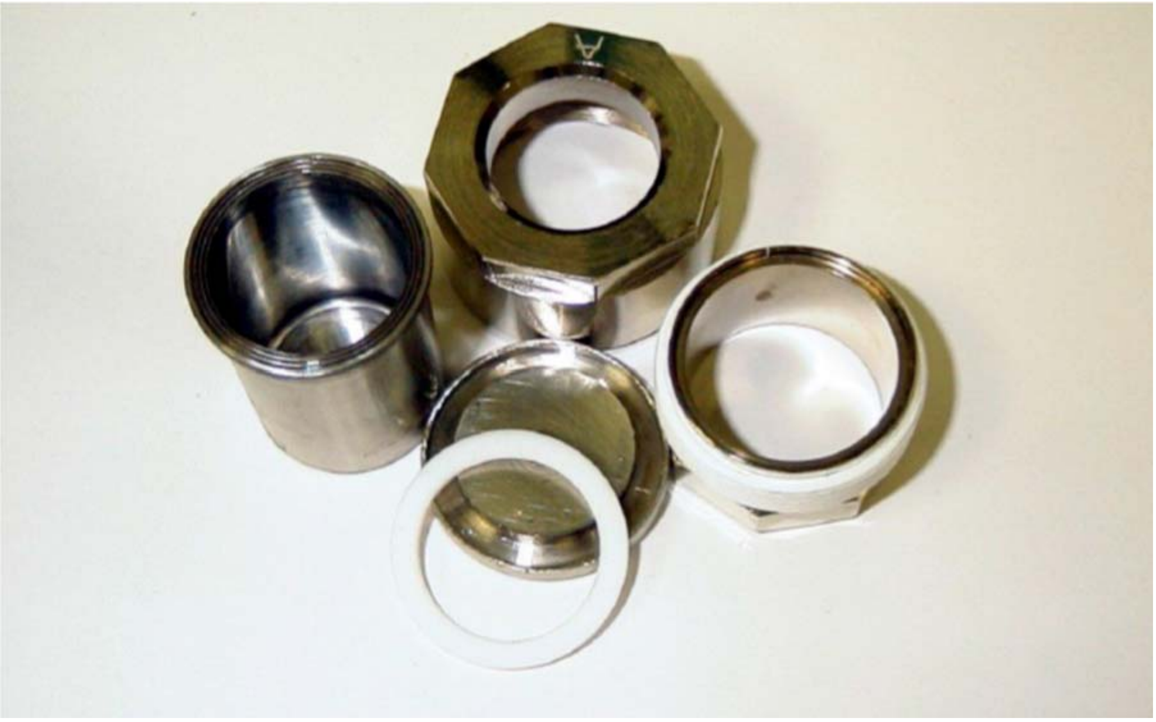

FIG. 1 (a) The Desensitized Type 304L Stainless Steel, 22 mL; PCT Vessel and Lid; a White Polytetrafluoroethylene Gasket Which Seals the Vessel and Lid When the Assembly is Closed and Tightened; and the Nickel-Plated Brass, Nut, and Screw Vessel Tightening Assembly.

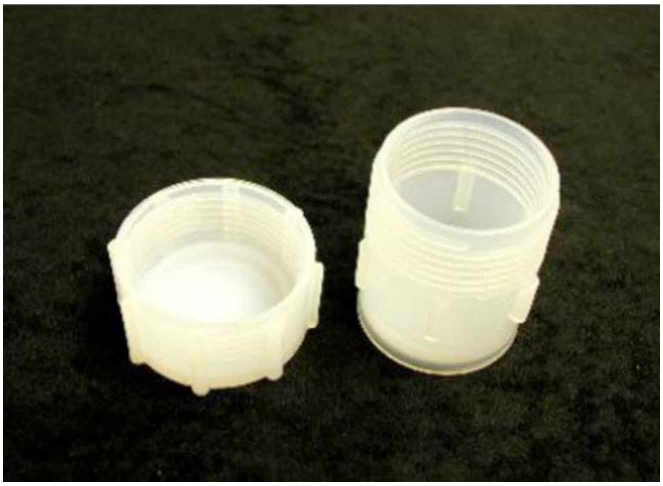

FIG. 1 (b) The 60 ml Polytetrafluoroethylene Vessel and Lid. The Vessels Should be Purchased with a Special Set of Plastic Wrenches (Not Shown) for Tightening. *(continued)*

can be expressed in terms of an average glass dissolution rate over the 7-day test 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).

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 of U.S. Standard ASTM − 100 to + 200 mesh (0.149–0.074 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 (0.420 mm). The particles are cleaned of adhering fines (see Note 1), and an amount of sized and cleaned glass waste form greater than or equal to 1 g is placed in either a Type 304 L stainless steel vessel or a PFA TFE-fluorocarbon vessel. An amount of ASTM Type I water equal to 10 ± 0.5 cm^3/g of sample mass (m_{solid})[5] 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 ± 2°C. Other test temperatures are permissible. It is desirable that intercomparison 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 ± 3.4 h, or other optional 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 1—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, ratios of sample-surface area (S) to leachant volume (V) (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 1×10^5 rad of beta or gamma radiation have been shown (**4**) not to damage TFE-fluorocarbon. If higher radiation doses are anticipated, degradation of the TFE-fluorocarbon gasket can compromise the seal and contaminate the leachate with 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 (>1×10^5 rad of beta or gamma radiation) will not generally be generated by hazardous, mixed or simulated nuclear waste glass forms. PFA TFE-fluorocarbon vessels, available commercially, can be used in the absence of high radiation fields[6] since PFA TFE-fluorocarbon is chemically inert when properly cleaned (**6**).

6.3 *Constant Temperature Devices*—Laboratory ovens or water baths capable of maintaining ±2.0°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 ±10°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.

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 laboratoryware.

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

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[6] PFA TFE-fluorocarbon is perfluoroalkoxy TFE-fluorocarbon. Labware of PFA TFE-fluorocarbon is manufactured by Savillex Corp., 6133 Baker Rd., Minnetonka, MN 55345 without plasticizers or organic additives.

6.12 *Syringes and Syringe Filters*—Sterilized, precleaned, or individually packaged syringes and mono or bidirectional 0.45 µm syringe filters.[7]

6.13 *Sample Vials*—Precleaned 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.*

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 colorometric 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 (see Ref **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_3) and hydrofluoric acid (HF) for cleaning leach vessels.[8]

8.2 *ACS High Purity Acid*—Ultra high purity concentrated nitric acid (HNO_3) for acidification of leachates.[8]

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,[9] then through a cellulose ester membrane having openings not exceeding 0.45 µm.[10]

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^3 (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.[8] All 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°C 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.

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[7] Cellulose acetate filters such as Nalgene No. 190-2045, available from Nalgene Co, or Gelman Sciences Supor Acrodisc 25, No. 4614 filters, available from Gelman Sciences, 600 S. Wagner Rd., P.O. Box 1448, Ann Arbor, MI 48106-9982, have been shown not to contaminate test solutions

[8] Available from American Chemical Society, 1155 16th St., NW, Washington, DC 20036.

[9] 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 ration, such as that available from the Millipore Corp., Bedford, MA 01730; Barnstead Co., 225 Rivermoor St., Boston, MA 02131; Illinois Water Treatment Co., 854 Cedar St., Rockford, IL 61105; or Vaponics, Inc., 200 Cordage Park, Plymouth, MA 02360, is suitable. 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,[1] which you may attend.

[10] An in-line filter such as those made by the Millipore Corp., Bedford, MA 01730; Gelman Instrument Co., 600 S. Wagner Rd., Ann Arbor, MI 48106; and Schleicher and Schuell, Inc., 540 Washington St., Keene, NH 10003, has been found to be satisfactory. 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,[1] which you may attend.

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_2 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_2 or O_2 into the leachate is not desired.

10.1.2 It is recommended that 22 mL steel vessels[11] be used for the radioactive production application in Test Method A (see Fig. 1a). This allows for the use of up to 1.8 cm^3 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 of $\leq$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 F from the vessel and ingress of CO_2 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_2 or O_2 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_2 and O_2, 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_2 and O_2 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[6]. The use of PFA TFE-fluorocarbon vessels under radiation doses $>10^5$ R causes degradation of the PFA TFE-fluorocarbon, and subsequent release of F^- 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^- 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 through 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_3 (1 weight % HNO_3) and heat to 90 ± 10°C for a minimum of 1 h.

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 ± 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 ± 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 through 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 through 12.1.9, then repeat the cleaning method starting at 12.1.4.

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[11] Vessels from Parr Instrument Co., 211 53rd St., Moline, IL 61265, have been found satisfactory. 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,[1] which you may attend.

12.1.12 Dry vessels and lids at 90 ± 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 ± 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 ± 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_3 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_3 (1 weight % HNO_3). Reseal the vessel and place in 90 ± 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_3 during cleaning or acid stripping of the vessel may be compromised because small amounts of HNO_3 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 ± 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^3 in the solution by methods such as ICP or Test Method D859, repeat 14.1.3 through 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^3 criteria cannot be achieved by three repetitions of 14.1.3 through 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 ± 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_3 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 to 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_3 during cleaning or acid stripping of the vessel may be compromised because small amounts of HNO_3 may be trapped between the gasket and the lid.

15.1.3 Soak the vessels and lids in 0.16 M HNO_3 (1 weight % HNO_3) 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^3 in the solution by methods such as ICP or Test Method D859, repeat steps 15.1.4 through 15.1.7.

15.1.9 If the 5.0 to 7.0 pH range or the >0.1 g/m^3 silicon criteria cannot be achieved after three repetitions of 15.1.4 through 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^- concentration of the water in each vessel by measuring the F^- concentration of another aliquot of the water from each vessel.

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

16.1.14 If the pH is above 7.0 repeat steps 16.1.7 to 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_3 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_3 (1 weight % HNO_3) 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^- concentration by measuring the F^- 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^- 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^- contamination.

17.1.8 If the pH is <5.0 or the F^- 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^- 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*—Samples must be ≥ 1 g. The reference ratio of leachant volume to sample mass (V_{soln}/m_{solid}) is 10 ± 0.5 cm^3/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 in 15 mL 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 Section 6.8).

19.5.2 Clean the brass or stainless steel sieves, catch pan and lid before and after every use (see Note 2).

NOTE 2—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 (see Note 3)). 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 (0.149 mm) sieve. A200 mesh (0.074 mm) sieve should be under the 100 mesh sieve with a catch pan below.

NOTE 3—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[12] shall produce a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample 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.

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[12] The Ro-Tap Testing Sieve Shaker, supplied by W.S. Tyler Co., Mentor, OH 44060, is suitable. 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,[1] which you may attend.

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.

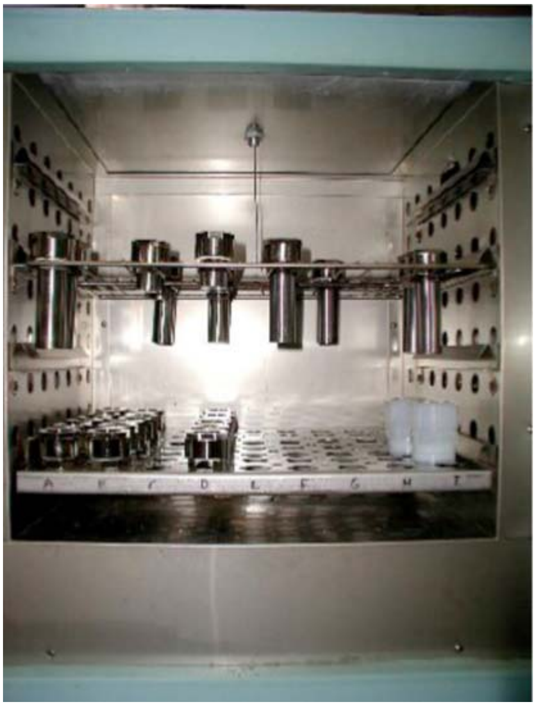
19.5.8 If additional material is needed, crush the + 100 mesh size waste form fragments again or begin again at step 19.5.1. After new waste form fragments have been prepared, repeat 19.5.3 through 19.5.7.

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 (74 µm), 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 through 19.6.8 should be omitted from the test procedure and noted on the data sheet such as that given in Appendix X2. If the separated phases are known to be water soluble, steps 19.6.9 through 19.6.11 must be performed in fresh absolute ethanol or another non polar solvent like cyclohexane. Any changes in the washing procedure steps 19.6.3 – 19.6.11 used should be noted on the data sheet such as that given in Appendix X2. Note that water washing is normally performed first when soluble secondary phases are not present in order to minimize generation of highly radioactive EPA hazardous



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.

solvents. It may be beneficial to remove fines by washing with absolute ethanol or another non polar solvent like cyclohexane and then quantify the exposed soluble phases by washing with water and analyzing the wash solutions.

19.6.2 Place sieved waste form in a clean glass beaker that will hold at least 2.5 times the sample volume. For example if 15 to 20 g of sieved sample is used, a 50 mL glass beaker should be used.

19.6.3 Forcibly add ambient temperature ASTM Type I water from a squirt bottle to the glass waste form in the beaker. The volume of water added should be about one and a half times the sample volume estimated from its height in the beaker. During water addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass. Swirl beaker for about five seconds to suspend the particles in water.

19.6.4 Allow the sample-water mixture to settle approximately 15 s, then decant off the water and discard.

19.6.5 Repeat 19.6.3 to 19.6.4.

19.6.6 Repeat 19.6.3.

19.6.7 Prepare the ultrasonic cleaner by filling with water to approximately 1 cm. Place the beaker from 19.6.6 in the ultrasonic cleaner for 2 min. After removing the beaker from the cleaner, decant the water from the beaker and discard.

19.6.8 Repeat 19.6.3 and 19.6.7.

19.6.9 Forcibly add absolute ethanol from a squirt bottle to the waste form. The volume of alcohol added should be about one and a half times the sample volume estimated from its height in the beaker. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass. Swirl beaker to suspend the particles in ethanol or another non polar solvent like cyclohexane.

19.6.10 Place the beaker from 19.6.9 in the ultrasonic cleaner for 2 min. After the 2 min, allow to settle about 15 seconds then decant the alcohol from the beaker and discard.

19.6.11 Repeat 19.6.9 through 19.6.10 two more times or until no suspended particles are visible in the ethanol or another non polar solvent like cyclohexane.

19.6.12 Put the beaker containing the cleaned sample in a 90 ± 10°C oven overnight to dry. If possible, examine the dried sample under a microscope to ensure that the fines have been removed and that there are no metal filings from the grinding admixed. Use a magnetic tweezers to move the sample particles around while observing the sample under the microscope. If metal filings from the grinding device are present they will stick to the magnetic tweezers making identification easier. If metal filings are present as contaminants from the grinding device, the sample must not be used. However, if the magnetic or metallic species, or both, are incorporated in the glass waste form do not discard the sample and proceed to 19.6.13.

19.6.13 Store the dried acceptable waste form in a clean, sealed and labeled container in a dessicator until use. Use within three months. If the samples have not been stored in a desiccator or they have been stored in a dessicator for more than three months, they must be redried at 90 ± 10°C overnight. Drying the sample before weighing ensures that the powders do not contain adsorbed water when weighed and therefore ensures that more accurate mass measurements and surface area measurements will be achieved for samples prepared at different times.

19.6.14 Enter sample identification, date, and name of the person performing the sample washing on a sample log sheet like that given in Appendix X2.

19.6.15 Periodically the efficiency of washing the fines off the meshed and washed sample should be checked by scanning electron microscopy as shown in Fig. 3.

20. Preparation of Reference Waste Form for PCT Test Method A

20.1 The reference waste form should be prepared at the same time as the waste forms are being tested. The same person should prepare the reference waste form using the same equipment that is used for the waste forms being tested. Use of the reference waste form provides confidence that the test is being performed correctly. If the reference waste form does not give the expected response, then the sievings or weighings, or both, or other operational steps may be in error and the test(s) must be repeated.

20.1.1 *Reference Waste Form Handling*—same as 19.1.

20.1.2 *Reference Waste Form Sample Size*—same as 19.3.

20.1.3 *Number of Standard Reference Waste Form Replicates*—same as 19.4.

20.1.4 *Crushing, Sieving, and Washing of Reference Waste Forms*—same as 19.5 and 19.6.

21. Procedure—PCT Test Method A

21.1 *Number of Sample Replicates*—All tests for each glass waste form should be carried out at least in triplicate (see 19.4).

21.2 *Number of Reference Glass Waste Form Replicates*—Tests with a standard glass shall be run at least in triplicate as part of each set of samples.

21.2.1 A set of samples is considered to be those that are tested simultaneously in the same oven.

21.3 *Number of Vessel Blanks*—A blank is considered to be a cleaned test vessel that has been filled with the same amount of ASTM Type I water that is used in tests with the waste form. For each set of samples, two blank tests conducted in vessels from the same batch of cleaned vessels shall be used. If more than one batch of cleaned vessels is used in tests with a set of samples, then blanks will be conducted in two vessels from each batch.

21.3.1 Enter batch cleaning identifier for the blanks, the blank vessel number, the date of blank cleaning, and the name of the person who cleaned the vessels for each sample on a sample log sheet like that given in Appendix X2.

21.4 *Testing Method*—Each sample, standard, and blank shall be tested according to the following method:

21.4.1 Collect a sufficient amount of fresh ASTM Type I water from the water purification system to fill all the leach vessels in the set of samples, including standards, and blanks being tested. Ensure that the Type I water meets the minimum

FIG. 3 Adherent fines on – 100 to + 200 mesh glass particles before washing (Top). Glass particles after washing in ASTM Type I Water and Absolute Ethanol (Bottom)

electrical resistivity of 18 MΩ·cm at 25°C. 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^3 (see Specification D1193 and Terminology D1129). Record the resistivity of the water collected on each log sheet. Record the resistivity of the water used for each sample waste form being tested, each reference waste form, and each blank.

21.4.2 Calibrate the pH meter. Determine the pH of the aliquot of ASTM Type I water collected in 21.4.1. Pour the water into a sealed cleaned vessel for transport to the shielded cell. Keep the water container sealed until use. Slow absorption of gaseous species from the air can cause the conductivity and pH of Type I water to slowly drift with time (consult Test Method D1293). Record the initial measured pH, the temperature at which the pH was measured, the pH values of the calibration solutions used, and the identification number of the pH meter used on a sample log sheet like that given in Appendix X2. Initial all measurement entries on the sheet. Discard the aliquot of ASTM Type I water used for the pH measurement.

21.4.3 Standardize the balance according to 18.2.2. It is recommended that a user's log of the balance standardization be kept. Record the balance identification number and the annual calibration date.

21.4.4 Weigh the empty vessel with the lid and gasket. Record the vessel number and the initial vessel weight on a sample log sheet like that given in Appendix X2.

21.4.5 Place the desired amount of prepared glass waste form in the clean vessel. The waste form may be preweighed prior to placing it in the vessel. If the waste form is preweighed, record the weight of the waste form. Replace the lid and reweigh the test vessel, lid and sample. Record this composite weight. If the waste form has not been preweighed prior to placing it in the vessel then the difference between the two vessel weighings should be recorded as the weight of the waste form on a sample log sheet.

21.4.6 Add a volume of ASTM Type I water equivalent to ten times the mass of glass waste form added as calculated in 21.4.5 so that $(V_{soln}/m_{solid}) = 10 \pm 0.5$ mL/g. Swirl to wet the glass waste form. Cap and seal the test vessel and reweigh. Record the total vessel weight on a sample log sheet. For blanks, add a similar amount of water to the vessel but no sample.

21.4.7 The set of samples including the reference waste form vessels, and blanks should be placed immediately into a preheated 90 ± 2°C oven. The seven day ± 2 % test period starts at this time. Record the date and time (d:h:min) and note on a sample log sheet.

21.4.8 The testing period shall be controlled to within ± 2 % of the total time period of the test, adjusting for any seasonal time changes as necessary. At the conclusion of the test remove the leach container from the oven. Record the date and time (d:h:min) at which the vessel is removed and note on a sample log sheet. The weighing, leachate pH measurement, and filtration in 21.4.9 to 21.4.12 must be done as soon as the vessel has cooled to ambient temperature.

21.4.9 Check the balance calibration according to 18.2.2. Record the balance identification number and the annual calibration date. It is recommended that a user's log of the balance standardization be kept. Weigh the cooled leach container plus contents. Record final weight on the sample log sheet and initial the entry. If the mass loss is calculated to be greater than 5 % of the original leachant mass, disregard the results of that test and use the remaining test results (minimum of two). If more than one replicate of the same glass waste form shows a mass loss of greater than 5 % of the original leachant mass, disregard the triplicate tests and the test specimens. Repeat the test in triplicate starting with new test specimens.

21.4.10 For remote operation with radioactive glass waste forms, the leachate may need to be removed from the radioactive cell to minimize contaminating the solution. In that case, carefully decant the leachate into a clean transport vessel and transfer this vessel to a radiochemical hood. Once this vessel is in the radiochemical hood, carefully transfer the solution from the transport vessel to another clean vessel to avoid contamination.

21.4.11 Calibrate the pH meter at the same temperature as the leachate being measured (see 18.2.3 for frequency). Pipette an aliquot of leachate into a clean, disposable container. Determine the pH of the aliquot after letting it cool to ambient temperature before you measure the pH. Record the measured pH as the final test pH on a sample log sheet. Record the pH values of the calibration solutions used, and the identification number of the pH meter used on a sample log sheet. Initial all entries. Discard the aliquot used for the pH measurement.

21.4.12 Draw a sufficient amount of the remaining leachate through a 0.45 µm syringe filter into a clean oil free disposable syringe (**2**, **25**). Note the usage of bidirectional filters allows the leachate to be filtered in either direction. Monodirectional filters are allowed but care must be exercised to filter in one direction only. More than one syringe filter may be necessary per sample. Transfer the filtrate into a clean specimen bottle for cation analysis (see Note 4). For leachates with pH values ≥11, the solution will need to be diluted before acidification (21.4.14) in order to prevent gellation of the leachate when acidified. If optional anion analyses are desired draw another filtered aliquot of the sample and transfer the contents of the syringe into a clean specimen bottle. Samples for anion analysis are not acidified (21.4.15).

NOTE 4—Sample vials can be cleaned by boiling specimen bottles and caps for 1 h in ASTM Type I water. Allow the specimen bottles to remain in the water overnight but reduce the temperature so that boiling has stopped. Remove the bottles and caps and dry in an oven at 80°C.

21.4.13 The solution analysis is discussed in Section 26.1, and should be consulted at this time.

21.4.14 Acidify the leachate aliquots drawn for cation analysis with concentrated ultra high purity HNO_3 equal to 1 volume % of the leachate aliquot volume (see Note 5). Perform cation analyses and include acidified multielement solution standards. For radioactive glass waste forms, submit appropriately acidified aliquots for all desired radiochemical analyses. Note the analytic service identification number (if any) on the sample log sheet along with the analysis requested.

NOTE 5—Other HNO_3 acidification/dilution techniques can be used if necessary; the final diluted sample should contain at least 1 % HNO_3 to prevent possible hydrolysis of heavy metal cations.

21.4.15 Anion analysis is optional but is strongly recommended. Use the non-acidified aliquot (see 21.4.12) for anion analysis. Analyze an unacidified multielement solution standard at the same time. Note the analytic service identification number (if any) on a sample log along with the analysis requested.

21.4.16 Measure cation and optional anion concentrations of glass waste form leachates, standard glass waste form leachates, blanks, and multielement solution standards (consult Test Methods C1109 and D4327).

21.4.17 Analysis of the solids on the filter or the remaining solid glass waste form sample is optional. If solids analysis is not desired, the filter and solid glass waste form sample may be discarded. If solids analysis is desired, record the appearance of the specimen particles, for example, visible changes in color, agglomeration, and physical characteristics. Wash the specimen particles from the vessel with pure water onto a clean watch glass and dry at 88 to 92°C. A temperature of 88 to 92°C will only drive off adsorbed moisture and not water of hydration. After drying, store in a clean container or analyze (for example, consult Practices C1463).

PRODUCT CONSISTENCY TEST (PCT)—TEST METHOD B

22. Sample Preparation for PCT Test Method B

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

22.2 *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 colled, have a visibly different appearance, or have cooled against a metal container (consult Appendix X1 of Guide D5956). The glass does not have to be annealed. Flush the sample surface with ASTM Type I water to remove potential surface contamination and dry before crushing.

22.3 *Choice of Sample Mass*—Samples must be ≥ 1.0 g. The recommended ratio of leachant volume to sample mass (V_{soln}/m_{solid}) is 10 ± 0.1 mL/g. Other V_{soln}/m_{solid} can be 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 in 15.0 ± 0.15 mL of leachant contained in a 22 mL steel vessel.

22.4 *Number of Sample Replicates*—A minimum of three tests with replicate samples shall be used to provide a measure of the experimental variance.

22.5 *Crushing and Sieving Glass Waste Forms*—If the glass waste form is redox sensitive, the user is cautioned that grinding the glass 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 **23**). 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. A representative sample of the material to be tested shall weigh at least twice the required weight of the amount of sample needed to perform the PCT in triplicate. It is recommended that the amount of the material prepared shall weigh at least twice the required weight of sample needed to perform the PCT in triplicate in order to have enough sized sample to test.

22.5.1 Transfer glass 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 Types 304L or 316L stainless steel because of the known interactions of mild steels and glass 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 steel, replace the sample basket as a precaution so that the sample does not contain excess iron.

22.5.2 Clean the brass or stainless steel sieves, catch pan, and lid before and after every use (see Note 6).

NOTE 6—It is recommended that brass or stainless steel sieves be cleaned by flushing with deionized water from all directions. Dry immediately with high pressure air or in an oven. Do not use solvents or high temperatures on brass sieves as the brass mesh has a protective film to inhibit corrosion.

22.5.3 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 glass waste form should be placed on an 8 in. diameter sieve (see Test Methods C225 and C371). (See Note 7.) Scaling the amount of glass 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 the largest mesh sieve. The smallest mesh chosen should be under the largest mesh sieve with a catch pan below. The recommended mesh sizes are 100 mesh (0.149 mm) and 200 mesh (0.074 mm).

NOTE 7—The variability of the sample surface area to the solution volume has the largest effect on the precision of the test (**25, 26**).

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

22.5.4.1 *Mechanical Sieving*—The mechanical shaking device[12] shall produce a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample 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).

22.5.4.2 *Hand Sieving*—Alternately tap and rotate the 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.

22.5.5 Remove the largest mesh size glass waste form fraction. Then remove the smallest mesh sieve containing the mesh fraction to be used in the test.

22.5.6 Tap the smallest mesh sieve forcefully over colored paper. For example, if the glass waste form is light colored, tap the sieve on dark paper. For a dark colored waste form, such as, black simulated 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).

22.5.7 Transfer the fraction of the sieved glass waste form to be used for testing 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.

22.5.8 If additional material is needed, recrush the waste form fragments of glass lying on top of the largest sieve screen or repeat 22.5.1. When new waste form fragments have been prepared repeat 22.5.3 through 22.5.7.

22.5.9 *Wet Sieving*—If the sample is friable it may be best to wet sieve with water, absolute ethanol, or another non polar solvent like cyclohexane. Water can be used if there are no soluble phases in the sample by forcefully squirting the sample with the liquid while the sample is on the sieve and over a catch vessel. 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 (74 µm), where powder surfaces develop enough of a surface charge and show an enhanced tendency to agglomerate or to minimize dusting from radioactive samples. (**27**)

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

22.6 *Washing the Sized Glass Waste Form:*

22.6.1 Washing of the size meshed glass waste form to remove adhering fines is required for homogeneous glass waste forms containing no secondary phases (see Fig. 3). 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 22.6.3 – 22.6.8 should be omitted from the test procedure and noted on the data sheet such as that given in Appendix X2. If the separated phases are known to be water soluble, steps 22.6.9 – 22.6.11 can be performed in fresh absolute ethanol or another non polar solvent like syclohexane. Any changes in the washing procedure steps 19.6.3 – 19.6.11 should be noted on the data sheet such as that given in Appendix X2. Note that water washing is normally performed first when soluble phases are not present in order to minimize generation of radioactive EPA hazardous solvents. It may be beneficial to remove fines by washing with absolute ethanol or another non polar solvent like cyclohexane and then quantify the exposed soluble phases by washing with water and analyzing the wash solutions.

22.6.2 Place sieved glass waste form in a clean glass beaker that will hold at least 2.5 times the sample volume. For example if 15 to 20 g of sieved sample is used, a 50 mL glass beaker should be used.

22.6.3 Forcibly add ambient temperature ASTM Type I water from a squirt bottle to the glass. The volume of water added should be about one and a half times the sample volume estimated from its height in the beaker. During water addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass. Swirl beaker for about five seconds to suspend the particles in water.

22.6.4 Allow the sample to settle approximately 15 s, then decant off the water.

22.6.5 Repeat 22.6.3 to 22.6.4.

22.6.6 Repeat 22.6.3.

22.6.7 Prepare the ultrasonic cleaner by filling with water to approximately 1 cm. Place the beaker from 22.6.6 in the ultrasonic cleaner for 2 min. After removing the beaker from the cleaner, decant the water from the beaker and discard.

22.6.8 Repeat 22.6.7.

22.6.9 Forcibly add absolute ethanol from a squirt bottle to the waste form. The volume of alcohol added should be about one and a half times the sample volume estimated from its height in the beaker. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates the entire sample. Swirl beaker to suspend the particles in ethanol or another non polar solvent like cyclohexane.

22.6.10 Place the beaker from 22.6.9 in the ultrasonic cleaner for 2 min. After the 2 min decant the alcohol from the beaker and discard.

22.6.11 Repeat 22.6.9 through 22.6.10 two more times or until no suspended particles are visible in the ethanol or another non polar solvent like cyclohexane.

22.6.12 Put the beaker containing the cleaned glass in a 90 ± 10°C oven overnight to dry. If possible, examine the dried sample under a microscope to ensure that the fines have been removed and that there are no metal filings from the grinding admixed. Use a magnetic tweezer to move the sample particles around while observing the glass under the microscope. If metal filings from the grinding device are present they will stick to the magnetic tweezer making identification easier. If metal filings are present as contaminants from the grinding device, the sample must not be used. However, if the magnetic or metallic species, or both, are incorporated in the glass waste form do not discard the sample and proceed to 22.6.13.

22.6.13 Store the dried acceptable waste form in a clean, sealed and labeled container in a desiccator until use. Use within three months. If the samples have not been stored in a desiccator or they have been stored in a desiccator for more than three months they must be re-dried at 90 ± 10°C overnight. Drying the sample before weighing ensures that the powders do not contain adsorbed water when weighed and therefore ensures that more accurate mass measurements and surface area measurements will be achieved for samples prepared at different times.

22.6.14 Enter sample identification, date, and name of the person performing the sample washing on a sample log sheet like that given in Appendix X2.

22.6.15 Periodically the efficiency of washing the fines off the meshed and washed sample should be checked by scanning electron microscopy as shown in Fig. 3.

23. Preparation of Reference Waste Form for PCT Test Method B

23.1 The reference waste form should be prepared at the same time as the glass waste forms that are being tested. The same sample mesh size, V_{soln} /m_{solid}, type of vessel, test duration, and test temperature as the samples being tested should be used. The same person should prepare the reference waste form using the same equipment that is used for the unknown glass waste forms being tested. Use of the reference waste form provides confidence that the test is being performed correctly. If the reference waste form does not give the expected response, then the sievings or weighings, or both, or other procedural step may be in error and the test(s) must be repeated.

23.1.1 *Reference Waste Form Handling*—Same as 22.1.

23.1.2 *Reference Waste Form Sample Size*—Same as 22.3.

23.1.3 *Number of Standard Reference Waste Form Replicates*—Same as 22.4.

23.1.4 *Crushing, Sieving, and Washing of Reference Waste Forms*—Same as 22.5 and 22.6.

24. Procedure—PCT Test Method B

24.1 *Number of Sample Replicates*—All tests for each glass waste form should be carried out at least in triplicate (see 22.4).

24.2 *Number of Reference Waste Form Replicates*—Tests with a standard glass shall be run at least in triplicate as part of each set of samples.

24.2.1 A set of samples is considered to be those which are tested simultaneously in the same oven for the same test duration.

24.3 *Number of Vessel Blanks*—A blank is considered to be a test conducted in a cleaned test vessel that has been filled with the same amount of ASTM Type I water or other leachant that is used in tests with a waste form. For each set of samples, two blanks tests conducted in vessels from the same batch of cleaned vessels shall be used. If more than one batch of cleaned vessels is used in tests with a set of samples, then blanks will be conducted in two vessels from each batch.

24.3.1 Enter batch cleaning identifier for the blanks, the blank vessel number, the date of blank cleaning, and the name of the person who cleaned the vessels for each sample on a sample log sheet like that given in Appendix X2.

24.4 *Testing Method*—Each sample, standard, and blank shall be tested according to the following method:

24.4.1 Collect a sufficient amount of fresh ASTM Type I water from the water purification system to fill all the leach vessels in the set of samples, including standards and blanks being tested. Ensure that the Type I water meets the minimum electrical resistivity of 18 MΩ·cm at 25°C. 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^3 (see Specification D1193 and Terminology D1129). If ASTM Type I water is used record the resistivity of the water collected on each log sheet. Record the resistivity of the water used for each sample waste form being tested, each reference waste form, and each blank. Other leachants can be used, including but not limited to simulated groundwater, actual groundwater, seawater, brine, and pH buffers. Add pertinent shelf life information on a log sheet like that given in Appendix X2.

24.4.2 Calibrate the pH meter. Determine the pH of the aliquot of the leachant collected in 24.4.1. Pour the leachant into a sealed cleaned vessel for transport. Keep the leachant container sealed until use. If ASTM Type I water is used note that slow absorption of gaseous species from the air can cause the conductivity and pH of Type I water to slowly drift with time (consult Test Methods D1293). Record the initial measured pH, the temperature at which the pH was measured, the pH values of the calibration solutions used, and the identification number of the pH meter used on a sample log sheet like that given in Appendix X2. Initial all measurement entries on the sheet. Discard the aliquot of leachant used for the pH measurement.

24.4.3 Standardize the balance according to 18.2.2. It is recommended that a user's log of the balance standardization be kept. Record the balance identification number and the annual calibration date.

24.4.4 Weigh the empty vessel with the lid and gasket. Record the vessel number and the initial vessel weight on a sample log sheet like that given in Appendix X2.

24.4.5 Place the desired amount of prepared glass waste form in the clean vessel. The waste form may be preweighed prior to placing it in the vessel. If the waste form is preweighed, record the weight of the waste form. Replace the lid and reweigh the test vessel, lid and sample. Record this composite weight. If the waste form has not been preweighed prior to placing it in the vessel, then the difference between the two vessel weighings should be recorded as the weight of the waste form on a sample log sheet.

24.4.6 Add a volume of leachant equivalent to the V_{soln}/m_{solid} chosen. A V_{soln}/m_{solid} of 10 ± 0.5 mL/g is the recommended equivalent to PCT-A. Swirl to wet the crushed glass waste form. Cap and seal the test vessel and reweigh. Record the total vessel weight on a sample log sheet. For blanks, add a similar amount of leachant to the vessel but no solid sample.

24.4.7 The set of samples including the reference waste form vessels, and blanks should be placed immediately into the oven preheated to the desired temperature. The temperature equivalent to PCT-A is 90 ± 2°C. The desired test period starts at this time. The test duration equivalent to PCT-A is seven days ± 2 %. Record the date and time (d:h:min) and note on a sample log sheet.

24.4.8 Leave sample vessels in the oven at test temperature between 4 to 16 h before testing the tightness of the lids. Quickly remove samples from the oven and retighten loose lids while the vessels are hot and return immediately to the oven for the remainder of the test duration. This is especially important when using PFA TFE-fluorocarbon vessels. Note which lids required additional tightening on a sample log sheet.

24.4.9 The testing period shall be controlled to within ± 2 % of the total time period of the test, adjusting for any seasonal time changes as necessary. At the conclusion of the test remove the leach container from the oven. Record the date and time (d:h:min) at which the vessel is removed and note on a sample log sheet. The weighing, leachate pH measurement, and filtration in 24.4.10 to 24.4.12 must be done as soon as the vessel has cooled to ambient temperature.

24.4.10 Check the balance calibration according to 18.2.2. Record the balance identification number and the annual calibration date. It is recommended that a user's log of the balance standardization be kept. Weigh the cooled leach container plus contents. Record final weight on the sample log sheet and initial the entry. If the mass loss is calculated to be greater than 5 % of the original leachant mass, the test is invalid. Disregard the results of that test and use the remaining test results (minimum of two). If more than one replicate of the same glass waste form shows a mass loss of greater than 5 % of the original leachant mass, disregard the triplicate tests and the test specimens. Repeat the test in triplicate starting with new test specimens.

24.4.11 Calibrate the pH meter at the same temperature as the leachate being measured (see 18.2.3 for frequency). Pipette an aliquot of leachate into a clean, disposable container. Determine the pH of the aliquot after letting it cool to ambient temperature before you measure the pH. It is important that the buffer solutions used to calibrate the pH meter and the test solutions be at the same temperature. Record the measured pH as the final test pH on a sample log sheet. Record the pH values of the calibration solutions used, and the identification number of the pH meter used on a sample log sheet. Initial all entries. Discard the aliquot used for the pH and temperature measurement.

24.4.12 Draw a sufficient amount of the remaining leachate through a 0.45 µm syringe filter into a clean oil free disposable syringe (**2, 25**). Note the usage of bidirectional filters allows the leachate to be filtered in either direction. Monodirectional filters are allowed but care must be exercised to filter in one direction only. More than one syringe filter may be necessary per sample. Transfer the filtrate into a clean specimen bottle for cation analysis (see Note 8). For leachates with pH values ≥11, the solution may need to be diluted with ASTM Type I water before acidification (24.4.14) in order to prevent gellation of the leachate when acidified. If optional anion analyses are desired draw another filtered aliquot of the sample and transfer the contents of the syringe into a clean specimen bottle. Samples for anion analysis are not acidified (24.4.15).

NOTE 8—Sample vials can be cleaned by boiling specimen bottles and caps for 1 h in ASTM Type I water. Allow the specimen bottles to remain in the water overnight but reduce the temperature so that boiling has stopped. Remove the bottles and caps and dry in an oven at 80°C.

24.4.13 Solution analysis is discussed in Section 26.1 and should be consulted at this time.

24.4.14 Acidify the leachate aliquots drawn for cation analysis with concentrated ultra high purity HNO_3 equal to 1 volume % of the aliquot volume (see Note 9). Perform cation analyses and include an acidified multielement solution standards. Note the analytic service identification number (if any) on the sample log sheet along with the analysis requested.

NOTE 9—Other HNO_3 acidification/dilution techniques can be used if necessary; the final diluted sample should contain at least 1% HNO_3 to prevent possible hydrolysis of heavy metal cations.

24.4.15 Anion analysis is optional but is strongly recommended. Use the non-acidified aliquot (see 24.4.12) for anion analysis. Analyze an unacidified multielement solution standard at the same time. Note the analytic service identification number (if any) on a sample log along with the analysis requested.

24.4.16 Measure cation and optional anion concentrations of glass waste form leachates, standard glass waste form leachates, blanks, and multielement solution standards (consult Test Methods C1109 and D4327).

24.4.17 Analysis of the solids on the filter or the remaining solid glass waste form sample is optional. If solids analysis is not desired, the filter and solid glass waste form sample may be discarded. If solids analysis is desired, record the appearance of the specimen particles, for example, visible changes in color, agglomeration, and physical characteristics. Wash the specimen particles from the vessel with pure water onto a clean watch glass and dry at ≤90± 2°C. A temperature of 90 ± 2°C will only drive off adsorbed moisture and not water of hydration. After drying, store in a clean container or analyze (for example, consult Practices C1463).

25. Calculation

25.1 *Calculation for Test Methods A and B:*

25.1.1 *Use of Multielement Standard*—Calculate the mean of the triplicate analyses and standard deviation of the analytic results of the multielement standard. If the mean values agree within 10 % of the standard values and the relative standard deviation is <10 %, then the analytic results are considered acceptable.

25.1.2 *Use of Blanks*—Blanks are used to determine if significant amounts of those elements which are in the glass waste form leach from the vessels (probably due to improper cleaning) or are present in the original leachant (whether the leachant is ASTM Type I water or a groundwater). An element is detected in solution when its concentration exceeds a critical level determined in part by the instrument being used and the solution matrix. The detection limit is commonly defined as the critical level plus three standard deviations from replicate analyses to account for random errors. A higher limit of the critical level plus 10 standard deviations is used to define quantitative analysis. A blank element concentration is considered significant if it is >10 % of the concentration of that respective element in the leachate of a test conducted with the waste form material. The mean concentration in the replicate blanks is used as a background concentration for all the sample leachate analyses for tests conducted in vessels from the same batch as the vessels used in the blank tests. If background corrections of >10 % of any element occur, data for that element are invalid.

25.2 *Calculations of Leachate Concentrations*—Leachate concentrations, especially those of the major soluble elements in the glass (see Section 26), are calculated for the waste form being tested and the standard waste form. Calculated values include corrections for loss of solution during the test and background concentrations; normalization to the glass composition and glass surface area. The leachate concentrations and pH values from tests with the standard waste form should be control charted. A control chart is a specific kind of run chart that allows significant change in the PCT response to be differentiated from the natural variability of performing the test . If the chart indicates that the PCT response being monitored is not in control, analysis of the chart can help determine the sources of variation (poor sieving, leaking vessels, poor analyses) which can then be eliminated to bring the PCT response back into control. Concentrations from each new set of analyses for the standard should be compared to previous test responses. If the current standard data is within the control chart range then all of the leachate test data generated at the same time as the standard was analyzed can be compared to previous data sets. If the data is outside the normal control chart range for the standard, then the data for all the samples must be bias corrected before it can be used. The bias corrections are defined as the differences between the current results and the control chart values. Use of a standard waste form allows the inter-laboratory and intra-laboratory long term and short term variables to be bias corrected. Use of the standard glass also allows data from different laboratories to be compared on an equivalent basis by bias correction of the inter-laboratory variables (**15**).

25.2.1 Calculate the final leachate volume for each test including blanks. The mass of water loss is equal to the weight loss that occurred during the test. The volume of water loss is equal to the mass water loss divided by the density of water, which can be assumed to be 1.00 g/cm^3: Vf=Vi – mass loss*density. Leachate losses of greater than 5 % of the initial volume invalidate Test Method A data (see 21.4.9). Leachate losses of greater than 5 % of the initial volume during Test Method B data should be investigated unless Test Method B is being used as a consistency test (24.4.10). All weight losses should be reported with the leachate data.

25.2.2 Calculate the average blank concentration in appropriate units for each element, by dividing the sum of the concentrations for the valid tests by the number of valid blank tests. The following equations can be used:

$$C_i(\text{blank}) = \left(\Sigma_{l=1}^{m} C_i(l) \cdot \frac{V_f(l)}{V_i(l)} \right) \cdot \frac{1}{m} \tag{1}$$

where:

C_i(blank) = average blank concentration, g_i/L,
$C_i(l)$ = concentration observed in the 1th blank, g/L,
$V_i(l)$ = initial volume of leachate blank, L,
$V_f(l)$ = final volume of the leachate blank, L,
m = number of replicate blank tests performed for vessels from the same cleaned batch, and
i = 1 to m.

The initial and final volumes are calculated from the measured initial and final masses using the density of the test solution, which can be assumed to be 1.00 g/cm^3 for demineralized water.

25.2.3 Calculate the standard deviation of the blank concentrations using Eq 2.

$$s = \sqrt{\frac{\sum_{l=1}^{m}\left[C_i(l) - C_i(blank)\right]^2}{m}} \tag{2}$$

where:

s = standard deviation of the blank concentrations,
$C_i(blank)$ = average blank concentration, g_i/L,
$C_i(l)$ = concentration observed in the ith blank, g/L, and
m = number of replicate blank tests performed for vessels from the same cleaned batch.

The standard deviation of the concentrations of an element measured in the replicate blanks is used to determine the significance of concentrations measured in tests with the material. To be deemed significantly different than the background, the concentration measured in the test with the material must be more than three standard deviations higher than the average concentration determined in the blanks from Eq 1 (greater than $C_i(blank) + 3s$).

25.2.4 Calculate the corrected leachate concentration for each sample replicate by subtracting the average blank concentration from the measured concentration (see Eq 3). For an element to be used as a measure of the extent of dissolution (for example, the test response), the concentration of that element measured in the test with material must be more than 10 standard deviations higher than the average concentration in the blank tests determined from Equation 1 (greater than $C_i(blank) + 10s$) of the leachate concentrations for a valid blank test. (**28, 29**)

If the blank concentrations for a given element are greater than three times the detection limit (**28, 29**) of the analytic method used for all replicate blanks, Eq 3 should be used to blank correct all calculations for that element. If the blank concentrations for a given element are less than three times the detection limit (**28, 29**) of the analytic method used for all replicate blanks, then the leachate concentrations do not have to be blank corrected as this may add error to the resulting leachate data rather than improve the leachate data. If the concentration of one element is high (greater than three times the detection limit (**28, 29**) in only one replicate blank, then all blank solutions from that cleaning batch need to be reanalyzed for elevated concentrations of that element since this may be an indication of a random vessel that may not have been adequately cleaned or a poor analysis on one of the vessels.

A decision must be made as whether to blank correct using the Eq 3 or not. It is recommended that the average blank concentration be used as the background concentration for concentrations high enough to provide a valid measure of the test response.

The following equation can be used to background correct each analysis:

$$C_i(\text{sample}) = \left(\frac{C_i(n)\cdot V_f(n)}{V_i(n)}\right) - C_i(\text{blank}) \tag{3}$$

where:

$C_i(n)$ = concentration observed in the test with the nth waste form sample, g/L,
$V_f(n)$ = final volume of leachate measured in the test with the nth waste form sample replicate, L,
$V_i(n)$ = initial volume of leachate in the test with the nth waste form sample replicate, L,
n = total number of replicate waste form sample tests performed, and
C_i(blank) = average blank concentration, g_i/L.

The initial and final volumes are calculated from the measured initial and final masses using the density of the test solution, which can be assumed to be 1.00 g/cm^3 for demineralized water.

25.3 Calculating the concentration in normalized units NC_i or log[NC_i] is recommended for all waste forms including multiphase waste forms wherein the concentrations in g/L are normalized by the weight fraction of that element present in the entire waste form. This provides an estimate of the total mass of material that goes into solution. Normalization is based on the entire waste form rather than the particular phase containing it because the value of NC_i is used to represent the entire waste form. Normalization to the waste form composition allows for direct comparison of NC_i measured for different waste forms. The normalized concentration, NC_i, is a function of (*1*) the mass fraction of the element of interest, *i*, in the glass and (*2*) the concentration of element *i* in solution.[13] The normalized concentration for each replicate is expressed as:

$$NC_i = \frac{c_i(\text{sample})}{f_i} \tag{4}$$

where:

NC_i = normalized concentration, $g_{\text{waste form}}/L_{\text{leachant}}$,
C_i(sample) = concentration of element "*i*" in the solution from test with waste form, g_i/L, and
f_i = mass fraction of element "*i*" in the unleached waste form (g_i/g_{glass}).

The units of NC_i for PCT-A are normally expressed as grams of glass waste form dissolved per litre of leachant when all of the tests are performed at the reference volume of leachant (V_{soln}) to sample mass (m_{solid}) ratio, for example, $V_{\text{soln}}/m_{\text{solid}}$ = 10 ± 0.5 cm^3/g, and with the 100 to 200 mesh reference particle size (see Appendix X1). Use of the reference conditions maintains the waste form surface area (A) to volume of

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[13] At the dilute solute concentrations utilized in this report, a 1 kg of solution is considered equivalent to 1 L of solution and so ppm and mg/L can be used interchangeably.

leachant (V) at a constant for waste forms with the same density. As long as the glass waste form density and glass waste form particle size remain the same between leach tests, this parameter will remain a constant and need not be calculated every time (see Appendix X1 for further discussion). Since triplicate PCT analyses must be performed with a glass waste form and composition is normally based on one or more analysis, there is not a one-to-one correspondence between the number of measurements that are averaged to determine the uncertainties in the solution (C_i) and the number of measurements that are averaged to determine the glass (f_i) concentrations. Thus the appropriate manner by which to compute, and thus report, the uncertainty in the normalized concentration term is as the antilog of the expression:

$$log(NC_i) = \log\,(\bar{C}\ i) - \log(\bar{f}\ i) \qquad (5)$$

where:

$log\,\bar{}\,(C\ i)$ = average of the three replicate logarithms of the corrected solution concentrations measured on a given sample,
$log\,\bar{}\,(f\ i)$ = average of the logarithms of the multiple glass concentrations measured on a sample, and
$log(NC_i)$ = logarithm of the uncertainty in C_i.

25.4 Normalization of the concentration to the surface area of the waste form used in the test allows for comparison of tests conducted at different SA/V ratios with materials having different densities, and test samples of different size fractions. For comparing the results of these tests, the geometric surface area is calculated by modeling the particles as spheres having a diameter equal to the arithmetic average of the sieve mesh sizes (Appendix X1). The specific surface area of a sphere having a particular diameter is calculated as

$$SA_{sp} = \frac{6}{\rho d} \qquad (6)$$

and the surface area is calculated as the product of the mass used in the test and the specific surface area of the material as

$$SA = \frac{6\ \ mass}{\rho d} \qquad (7)$$

where:

ρ = density in g/cm^3,
d = particle size diameter in cm,
$mass$ = mass in g,
SA = surface area in cm^2, and
SA_{sp} = specific surface area in g/cm^2

The average diameter for particles in the –100 +200 mesh size fraction is 112.5.

25.5 A normalized elemental mass loss, NL_i, can be calculated when the particle size, or the V_{soln}/m_{solid}, are varied between tests, or for test results on waste forms of different density. The following expression may be used to calculate NL_i:

$$NL_i = \frac{c_i(\text{sample})}{(f_i)\cdot(SA/V)}$$

$$or$$

$$NL_i = \frac{NC_i}{SA/V} \qquad (8)$$

where:

NL_i = normalized elemental mass loss, $g_{\text{waste form}}/\ m^2$,
c_i(sample) = concentration of element "i" in the solution, g_i/L,
f_i = mass fraction of element "i" in the unleached waste form (g_i/g_{glass}), and
SA/V = surface area divided by the leachate volume, m^2/L,

NL_i represents the total mass of glass dissolved over the test interval presuming all components dissolve congruently with element i and the surface area remains constant as the glass dissolves.

25.6 Calculate the standard deviation for the background corrected leachate concentrations for valid element concentrations.

26. Report

26.1 The most important elements to be analyzed in the leachate are those that best represent the extent of dissolution of the glass waste form. For example, elements that are not sequestered in altercation phases that participate in surface alteration reactions, and are also not solubility limited, 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 (**30-42**) of any glass or glass ceramic waste form must be performed in order to determine what elements represent the extent of dissolution of the glass waste form. This bounds the extent of dissolution of the radioactive constituents. For example, in high level borosilicate waste glass, Tc^{99}, present at ~4.1 × 10^{-4} weight % in the waste form, has been shown to be released congruently (stoichiometrically) with boron, lithium, and sodium. (**33**) Therefore, for borosilicate glass waste forms, the leachates are routinely analyzed for boron, lithium, and sodium if these elements are present at > 1 mass % in the glass. Additional mechanistic information about high level borosilicate waste glass durability is gained by analyzing for other elements present in the glass.

26.2 Report all results as the concentration of the elements in solution and as the concentration of the elements in solution normalized by the weight fraction of that element present in the glass waste form (see Section 25 and consult Practices C1463). The detection limits for each analysis must accompany the reported results.

26.3 All data should be recorded in a retrievable manner.

26.4 Deviations from the test method and the expected effect on the results should be justified in any reports.

27. Precision and Bias[14]

27.1 *Precision—Test Methods A and B (see Note 10):*

NOTE 10—Precision cited from Refs (**2** and **43**) are for Version 1.0 and 2.0 of the PCT which did not require sample washing. Better precision has been observed when samples are washed (**2 and 25**). In addition, the data in (**2** and **43**) used polytetrafluoroethylene vessels as this procedure had not been split into Test method A and B at the time these round robins were performed. These statistics are found in Table 2 as examples of how incorrect washing or no washing at all can impact the test results.

27.1.1 The measures of precision were determined in accordance with procedures in Practices E177 and E691. These measures are designated as follows:

27.1.1.1 *Repeatability*—The standard deviation for within-laboratory determinations.

27.1.1.2 *Reproducibility*—The standard deviation for between-laboratory determinations.

27.1.1.3 *%RSD*—The standard deviation divided by the consensus mean and multiplied by 100.

27.1.2 *PCT Method A: Within-Laboratory Precision*—A round robin was completed involving eight laboratories in order to make a statement concerning the repeatability for Test Method A. This round robin used the low-activity reference material (LRM) glass (**13 and 14**) (27.1.2.1). Data from two separate laboratories have been used to determine the within laboratory precision for remote radioactive operation (27.1.2.2 through 27.1.2.3). Within laboratory precision has also been determined for "hands on" operation with a non-radioactive glass (27.1.2.4).

27.1.2.1 *LRM Glass*—The within-laboratory precision of the eight laboratories based on the concentrations of silicon, sodium, and boron in the PCT leachates was established using washed -100 to +200 mesh glass in ASTM Type I water for seven days (data in Ref (**13, 14, and 44**)). Each laboratory performed triplicate tests on the LRM glass. For silicon the relative within laboratory precision was 1.52 %. For boron and sodium, the values were 2.42 and 2.54 %.

27.1.2.2 *Radioactive Glass 200R* (a radioactive borosilicate glass containing U-235 neutron fission and activation products)—Within-laboratory standard deviations for the measured boron concentrations (ppm) were 2.2 and 5.3 % using washed – 100 to + 200 mesh glass in ASTM Type I water for seven days (data in Ref **30**). For silicon the within laboratory relative standard deviations were 0.7 and 1.1 %. Each laboratory performed triplicate tests on each glass.

27.1.2.3 *Radioactive Glass 165/42* (a radioactive borosilicate waste glass containing U-235 neutron fission and activation products)—Within-laboratory standard deviations for boron were 2.9 and 3.5 % using washed – 100 to + 200 mesh glass in ASTM Type I water for seven days (data in Ref **30**). For silicon the standard deviation for both laboratories was 3.2 %. Each laboratory performed triplicate tests on each glass.

27.1.2.4 *Nonradioactive Glasses SRL 202-P, 202-G*—(borosilicate glasses containing simulated nonradioactive nuclear waste) and ARM-1, a simulated borosilicate nuclear waste glass (**43**), and SRM-623 a borosilicate glass NIST standard reference material, (approved reference materials)—Within laboratory relative standard deviations for boron for the four glasses were 3.0, 2.1, 8.1 and 2.0%, respectively (data in Ref **43**). The four non-radioactive glasses were tested three times in triplicate and each leachate was analyzed twice. For each glass there were eighteen (18) leachate analyses. Note that the precision of PCT-A performed remotely with radioactive glass is comparable to the precision presented above where the glass is handled directly.

27.1.3 *PCT Method A: Between-Laboratory Precision*—A round robin was completed involving eight laboratories in order to make a statement concerning the reproducibility for Test Method A. This round robin used the low-activity reference material (LRM) glass (**13, 14, and 44**) (see 27.1.3.1). Data from two separate laboratories have been used to determine the between laboratory precision for remote radioactive operation (see 27.1.3.2 through 27.1.3.3).

27.1.3.1 *LRM Glass*—The within-laboratory precision of the eight laboratories based on the concentrations of silicon, sodium, and boron in the PCT leachates was established using washed -100 to +200 mesh glass in ASTM Type I water for seven days (**13, 14, and 44**). Each laboratory performed triplicate tests on the LRM glass. For silicon, the relative between laboratory precision was 5.46 %. For boron and sodium, the values were 9.51 and 7.44 %.

27.1.3.2 *Radioactive Glass 200R* (a radioactive borosilicate glass containing U-235 neutron fission and activation products)—Between-laboratory relative standard deviations for boron and silicon were 13 % and 11 %, respectively, using washed – 100 to + 200 mesh glass in ASTM Type I water for seven days (Data in Ref **30**). Each laboratory performed triplicate tests on each glass.

27.1.3.3 *Radioactive Glass 165/42* (a radioactive borosilicate waste glass containing U-235 neutron fission and activation products)—Between-laboratory relative standard deviation for boron and silicon was 14 % using washed – 100 to + 200 mesh glass in ASTM Type I water for seven days ±3.4 h (Data in Ref **30**). Each laboratory performed triplicate tests on each glass.

27.1.4 *PCT Method B—Within-Laboratory Precision for Borosilicate Glass:*

TABLE 2 Summary of Measured Intralaboratory and Interlaboratory Precision for Common Reference Glasses Using Test Method A

Material	Intralaboratory Precision, %rsd				Interlaboratory Precision, %rsd			
	B	Li	Na	Si	B	Li	Na	Si
ARM-1[A] (unwashed)	7.53		7.48	4.89	12.11		9.14	7.52
SRM 623[A] (unwashed)	7.04		6.85	8.07	8.64		7.57	9.70
LRM[B] (washed)	2.42		2.54	1.52	9.51		7.44	5.46
EA[C] (washed)	7.33	7.37	6.75	9.63				
EA[D] (washed)	5.87	8.28	4.22	9.15				

[A] Results calculated from data in (**43**) for single tests run during weeks 1, 2, and 3 (from s_R from Table C.2 and s_r from Table C.3).

[B] Results from (**44**).

[C] Results from (**11, 16**).

[D] Results from (**22, 23**).

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[14] All precision and bias statements were calculated on leachate analyses in ppm (mg/L).

27.1.4.1 *Approved Reference Material* (ARM-1, a simulated borosilicate nuclear waste glass (**9**))—Within-laboratory relative standard deviation for boron was 2.3 % and for silicon was 1.8% using unwashed − 100 to + 200 mesh glass in ASTM Type I water for seven days (**2**). All tests were performed in PFA TFE fluorocarbon vessels in ASTM Type I water at the reference PCT-A conditions. Each of the three laboratories analyzed the glass in triplicate.

27.1.5 *PCT Method B—Within-Laboratory Precision for Multiphase Glass Ceramics:*

27.1.5.1 *Nonradioactive Glass Bonded Sodalite* (a glass-ceramic material consisting of ~25 wt % borosilicate glass, ~72 wt% sodalite, ~3 wt% halite, and other minor ceramic phases)—A round robin was conducted with four participating laboratories performing tests using TFE-fluorocarbon vessels at the reference PCT-A conditions. Two solutions were generated and evaluated during the round robin, the water wash solution (see 19.6.1) and the primary test solution (see 21.4.13). Only the interlaboratory precision of the primary test solution was evaluated. The intralaboratory relative standard deviation for the primary test solutions was 2.91 %. For boron and sodium, the values were 5.02 % and 10.43 % respectively. The %RSD for sodium in the water wash solutions from triplicate tests by a single laboratory were 9.55 % and 2.26 %. (**44**)

27.1.6 *PCT Method B—Between-Laboratory Precision for Borosilicate Glass:*

27.1.6.1 *Approved Reference Material* (ARM-1, a simulated borosilicate nuclear waste glass (**9**))—Interlaboratory relative standard deviation (including within laboratory and between laboratory % RSD) for boron was 12.1% and for silicon was 7.5% for laboratories with varying analytic capabilities. Unwashed − 100 to + 200 mesh glass in ASTM Type I water for seven days was used in the intra-laboratory comparison (Data in Ref **43**). Each of the six laboratories analyzed each glass in triplicate once a week for three weeks and each leachate was analyzed twice. Each of the six laboratory populations are, therefore, eighteen (18) test results.

27.1.6.2 *NIST Reference Glass SRM—623* (a borosilicate glass NIST standard reference material)—Between-laboratory standard deviation (including within laboratory and between laboratory % RSD) for boron was 8.6% and for silicon was 9.7% for laboratories with varying analytic capabilities. Unwashed − 100 to + 200 mesh glass in ASTM Type I water for seven days was used in the intra-laboratory comparison (data in Ref **43**). Each of the six laboratories analyzed each glass in triplicate once a week for three weeks and each leachate was analyzed twice. Each test population is eighteen (18) test results.

27.1.6.3 *Nonradioactive Glasses SRL 202-P, 202-G* (borosilicate glasses containing simulated nonradioactive nuclear waste)—Between laboratory relative standard deviations for boron for the SRL 202-P and 202-G were 5.0% for laboratories with varying analytic capabilities. The between-laboratory %RSD for silicon for SRL 202-P and SRL202-G were 3.4% and 3.7% respectively. Unwashed − 100 to + 200 mesh glass in ASTM Type I water for seven days was used in the intra-laboratory comparison (data in Ref **43**). Each of the six laboratories analyzed each glass in triplicate once a week for three weeks and each leachate was analyzed twice. Each test population is eighteen (18) test results.

27.1.7 *PCT Method B—Between-Laboratory Precision for Multiphase Glass Ceramics:*

27.1.7.1 *Nonradioactive Glass Bonded Sodalite* (a glass-ceramic material consisting of ~25 wt % borosilicate glass, 72 wt% sodalite, ~3 wt% halite, and other minor ceramic phases)—A round robin was conducted with four participating laboratories in TFE-flurocarbon vessels at the reference PCT-A conditions. Two solutions were generated and evaluated during the round robin, the water wash solution (see 19.6.1) and the primary test solution (see 21.4.13). For silicon, the interlaboratory relative standard deviation for the primary solutions was 9.11 %. For boron and sodium, the values were 15.4 % and 11.3 %, respectively. The %RSD for sodium and chloride in the water wash solutions from single tests at each laboratory were 7.32 % and 6.97 %. (**44**)

27.2 *Bias:*

27.2.1 For both PCT Methods A and B the average corrected leachate concentrations for the standard glass allow assessment of long term bias or variability of the test, for example, how reproducible the experimental variables such as oven temperature, sieving, leachate analyses, etc. are over time. Use of a standard glass allows the inter-laboratory long term and short term variables to be bias corrected. Use of the standard glass also allows data from different laboratories to be compared on an equivalent basis by bias correction of the intra-laboratory variables (**15**).

APPENDIXES

(Nonmandatory Information)

X1. SAMPLE CALCULATIONS OF THE EXPOSED GLASS WASTE FORM SURFACE AREA

X1.1 Discussion of Surface Area Measurement Techniques X1.1 In order to express the PCT response as a normalized release (see Eq 8), the surface area of the glass waste form exposed to the leachant must be determined. While surface area measurements can be geometric (using the average sieve size and glass density), or determined by particle size analysis (PSA), or determined by BET (Brunauer-Emmett-Teller) measurement methods, there are errors associated with each method. McGrail, et.al. (**45**) have stated that the geometric methodology of the determination of surface area as given in this test protocol (Test Methods C1285) is the correct way to assess the surface area of vitreous waste forms while BET surface area is the correct way to assess the surface area of ceramic/mineral waste forms in Single Pass Flow Through (SPFT; ASTM 1662) and Pressurized Unsaturated Flow (PUF;**46**) tests in order to account for the additional surface roughness in ceramic/mineral vs. glass waste forms. The measured, BET method more accurately determines the surface area of ceramic/mineral waste forms due to the more porous and irregular surface of these waste forms. (**47**) The surface of a glass waste form is much more regular (Fig. 3) than a ceramic/mineral waste form and the calculated geometric surface area from the arithmetic average of the sieve sizes is typically used for determining dissolution behavior.

Spherical glass bead standards are available from NIST (see discussion in Ref. **48**) for the calibration of various sieve sizes.

Reference **48** is a "NIST Recommended Practice Guide For Particle Size Analysis" that discusses the sources of error from Laser Light Diffraction Techniques used for particle size analysis. These include errors that are sample and preparation related and those which are instrument related. In terms of sample and preparation these errors include errors introduced due to use of non-representative samples and riffling of samples is suggested as the best method for homogenizing a powdered sample. Errors due to the analysis of powders finer or coarser than the detection limits of the instrument being used are discussed as are errors due to analysis of non-spherical powders. Any deviations from sphericity will cause bias and errors to be introduced in the particle size and size distribution results. Unless analyzed by appropriate algorithms designed as part of the instrument software, or designed for use on the obtained scattering patterns, it may be expected that the magnitude or error will be magnified as the deviation from spherical shapes is increased. Errors can also be associated with the optical properties of the material including the density of the material. Also, reliable procedures should be in place to maintain a stable dispersion of the powders in the sample cell since the instrument lacks the ability to distinguish between primary particles and agglomerates. Instrument errors can be introduced due to non-aligned or misaligned optics. Instrument errors can also be due to lack of background signals or errors in procedures for obtaining background signals, errors due to light leakage due to stray or extraneous light in the instrument will cause additional signals at the detector elements that will be analyzed as diffracted signals from the particles and be included in the size distribution results. The use of incorrect optical models will have a significant effect on the calculated size distributions. These effects may be manifest not only in the range of the size distribution, but also in the shape of the distribution. Software related bias may cause significant errors in the calculated results. Most errors would arise due to the design of the deconvolution and inversion algorithms that may be based on assumptions not reflected or applicable to the particle system under study. An example of such an error would be the use of a model-dependent inversion procedure (that is, inversion procedure that assumes a particular shape for the powder distribution) on a multi-modal powder system. Errors due to non-linear detector responses arise when sample loading is either too high or too low. Therefore, Microtrac should not be used to determine the average particle size for PCT analyses.

X1.2 Calculation of the Average Particle Size X1.2 Glass waste form particles are neither spherical, tabular, nor square (see Fig. 3). In order to calculate the exposed glass waste form surface area from the mass of glass waste form used, one must assume a given geometric shape. Because the density of the glass is used in the calculation of the exposed surface area, the density must be measured (by Test Method C693 or pycnometry) on a larger piece of glass before the glass is crushed and sized. A set of model calculations is given in Table X1.1 that assume spherical shaped particles (using Eq 6) to show the impact of particle size and glass density on the surface area (S) calculated. As long as the glass waste form density and glass waste form particle size and distribution remain comparable between leach tests, this parameter will remain a constant and need not be calculated every time. Assuming cubic (**49**) or tabular (**50**) shapes imparts only an approximately 1 % difference in the surface area calculation compared to assuming spherical shapes. For tabular shapes the error could be greater depending on the aspect ratio of the particles.

If the glass waste form is assumed to be spherical and the arithmetic average of the sieve size between the –100 (0.149 mm) to + 200 (0.074 mm) mesh is assumed to be 112 mm or 1.12×10^{-4} m, the following calculation allows an estimated glass surface area to be calculated.

The density of the particle is

$$\rho = \frac{mass}{volume} \tag{X1.1}$$

The specific surface area is given by

$$SA_{sp} = \frac{area}{mass} = \frac{\pi d^2}{\rho \times volume} = \frac{6\pi d^2}{\rho \times \pi d^3} = \frac{6}{\rho d} \tag{X1.2}$$

Also consult Eq 6 and 7 in the procedure.

Note that the specific surface area decreases as the density and particle size increase. The total surface area is the product of the specific surface area and the mass of glass.

TABLE X1.1 Variation of the Exposed Surface Area for 1 gram of Glass at Constants Mesh Sizes and Varying Densities.

Mesh Size	Known Glass of Density Shown	Sieve Micron Size Range	Average Sieve Particle Diameter (m)	Density (kg/m^3)	SA$_{GEO}$(m^2)	SA$_{GEO}$/V(m^{-1})
					for 1 gram	
100–200		150–74	1.12E-04	2.00E+03	2.67E-02	2667
				2.25E+03	2.37E-02	2370
	LRM			2.516E+03	2.10E-02	2100
	SRL-202			2.71E+03	1.97E-02	1968
	AFCI			2.70E+03	1.88E-02	1883
				3.00E+03	1.78E-02	1778
	LABS			3.56E+03	1.50E-02	1498
				4.00E+03	1.33E-02	1333

The surface area provided by the +200 mesh particles is much less than the surface area provided by the +200 mesh particles on a per mass basis: the surface area decreases 6-times as fast as the diameter (or density) increases. Based on the constant particle size and varying density shown in Table X1.1 and the relationship between the surface area and diameter of a spherical particle, the specific surface area calculated by modeling the sieved fraction as spheres with a nominal diameter is expected to provide a good estimate of the total surface area.

Recent tests were conducted to compare the dissolution rates measured with crushed particles (-100+200 mesh size fraction) in single-pass flow-through tests (ASTM C1662) and with polished monoliths in immersion tests (ASTM C1220) the same pH and temperature **(51)**. The rate calculated by assuming the particles had a specific surface area of 0.0210 m^2/g (the density of the glass was 2.516 g/cm^3) was only 1.5 % lower than the rate measured with monolithic specimens with directly-measured geometric surface areas. This suggests the estimated surface area of the particles was 1.5 % too high, but is well within the experimental uncertainty.

The normalized mass loss and normalized release can then be expressed in terms of test parameters in Eq X1.3, which gives the same results as Eq 8.

$$NL_i = \frac{C_i V_f \rho d}{6 f_i m} \tag{X1.3}$$

where:

NL_i = the normal elemental mass loss, $g_{waste\ form}/m^2$
C_i = the concentration of the element of interest, g_i/L
V_f = the final leachate volume, L,
r = the glass density, $g_{waste\ form}/cm^3$
d = the average of the mesh size fraction or diameter of the particle in cm,
f_i = the mass fraction of the element of interest in the waste form ($g_i/g_{waste\ form}$), and
m = the mass of waste form used in the test ($g_{waste\ form}$)

X2. MODEL PCT DATA SHEET

Sample ID					
Sample Preparation					
1.Ground/Sieved		Date		By	
2.Sample Washed		Date		By	
3.Sample Dried		Date		By	
Vessel Preparation					
1.Sample Vessel Cleaning		ID#		Batch cleaning #	
		Date		By	
2.1st Vessel Blank		ID#		Batch cleaning #	
3.2nd Vessel Blank		ID#		Batch cleaning #	
4.Blanks cleaned		Date		By	
Run Data					
	Initial Conditions	Persons Initials	Final Conditions	Persons Initials	Change
1.Type of solution	▨	▨	▨	▨	▨
2.Water meter ID# and date calibrated	▨	▨	▨	▨	▨
3.MΩ•cm Type I water at 25°C	▨	▨	▨	▨	▨
4.MΩ•cm calibration at 25°C	▨	▨	▨	▨	▨
5.pH/temp (°C) of leachant					
6.pH/temp (°C) of buffer solutions					▨
7. pH neter ID# and date calibrated					▨
8.wt of empty vessel+lid (gms)					▨
9. wt of vessel + lid + sample (gms)					▨
10.wt of sample (Item #9-#8)					▨
11.mL of solution Item #10*10					▨
12. wt of vessel + sample + lid + solution					Note X2.1
13. ID# of Balance and date calibrated					▨
14. Test (Revision #, Method A or B, and temperature		▨	▨	▨	▨
15.date, hour, min test started/ ended					Note X2.2
16. ID# of Oven and date calibrated					
17.Teflon Vessel lid retightened (Y/N)					
Leachate Analyses					
1. Acidified with		Date	By		
2. Diluted with		Date	By		
3. Dilution factor		Date	By		
REMARKS AND DEVIATIONS					

NOTE X2.1—If greater than 5 % of initial value for PCT Test Method A and B the data is not usable.

NOTE X2.2—If greater than 2 % of total time period then data is not usable.

Leachate Analyses:
Acidifed with ______________________________ on _______ by _______
Diluted with ______________________________ on _______ by _______
Diluted factor ______________________________ on _______ by _______

Analytic Service ID's:
Undiluted Cation Analysis No. ______________ for ____________________
Diluted Cation Analysis No. _______________ for ____________________
Corresponding Blank ID No. (Optional) _______ for ____________________

Undiluted Anion Analysis No. ______________ for ____________________
Diluted Anion Analysis No. ________________ for ____________________
Solids Analysis No. _______________________ for ____________________

Remarks/Deviations/Comments:

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