



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 C 1285; 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 evaluate the chemical durability of homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, and/or multiphase glass ceramic waste forms hereafter collectively referred to as “glass waste forms” by measuring the concentrations of the chemical species released to a test solution.

1.1.1 Test Method A is a seven-day chemical durability test performed at $90 \pm 2^\circ\text{C}$ in a leachant of ASTM-Type I water. The test method is static and conducted in stainless steel vessels. 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, mesh size, mass of sample, leachant volume, and leachant compositions. This test method is static and can be conducted in stainless steel or PFA TFE-fluorocarbon vessels, or both. Test Method B can specifically be used to evaluate the relative chemical durability characteristics of homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, and/or multiphase glass ceramic waste forms. This test method is applicable to radioactive (nuclear) and mixed, hazardous, and simulated 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:²

- C 92 Test Methods for Sieve Analysis and Water Content of Refractory Materials
- C 162 Terminology of Glass and Glass Products
- C 169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass
- C 225 Test Methods for Resistance of Glass Containers to Chemical Attack
- C 371 Test Method for Wire-Cloth Sieve Analysis of Non-plastic Ceramic Powders
- C 429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture
- C 693 Test Method for Density of Glass by Buoyancy
- C 1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy
- C 1174 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
- C 1463 Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis
- C 1125 Test Method for Penetration Index of Asbestos
- D 1129 Terminology Relating to Water
- D 1193 Specification for Reagent Water
- D 1293 Test Methods for pH of Water
- D 4327 Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

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 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.

- E 7** Terminology Relating to Metallography
- E 177** Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E 456** Terminology Relating to Quality and Statistics
- E 691** Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E 1402** Terminology Relating to Sampling

3. Terminology

3.1 Definitions:

3.1.1 *anneal*—to prevent or remove materials processing stresses in glass by controlled cooling from a suitable temperature (modified from Terminology **C 162**).

3.1.2 *annealing*—a controlled cooling process for glass designed to reduce thermal residual stress to an acceptable level, and, in some cases, modify structure (modified from Terminology **C 162**).

3.1.3 *ASTM Type I water*—purified water with a maximum total matter content including soluble silica of 0.1 g/m³, 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 **D 1193** and Terminology **D 1129**).

3.1.4 *chemical durability*—in these test methods, the resistance of a glass waste form to the release of its constituents to an aqueous solution under the specific conditions of this test.

3.1.4.1 *Discussion*—The response of a glass under other conditions is outside the scope of these test methods.

3.1.5 *closed system tests*—a system that precludes the transport of matter either into or out of the system.

3.1.6 *consistently controlled*—to verify with a high degree of accuracy, as an experiment, by comparison with a standard or a target, or by other experiments. (*Webster's New Twentieth Century Dictionary, 1973*)

3.1.7 *devitrified glass*—a homogeneous or phase (or both) separated glass that has partially crystallized during cooling or due to thermal heat treatment, or both.

3.1.8 *glass*—an inorganic product of fusion that has cooled to a rigid condition without crystallizing (see Terminology **C 162**); a noncrystalline solid or an amorphous solid.³

3.1.9 *glass ceramic*—solid material, partly crystalline and partly glassy (see Terminology **C 162**).

3.1.10 *hazardous waste glass*—a glass comprised of glass forming additives and hazardous waste.

3.1.11 *homogeneous glass*—a glass that is a single amorphous phase; a glass that is not separated into multiple amorphous phases.

3.1.12 *leachant*—the solution that is being used, or is intended for use, in a durability test.

3.1.13 *leachate*—the solution resulting from a durability test.

3.1.14 *mixed waste*—waste containing both radioactive and hazardous components regulated by the Atomic Energy Act (AEA) **(1)**⁴ and the Resource Conservation and Recovery Act (RCRA), **(2)** respectively; the term “radioactive component”

refers only to the actual radionuclides dispersed or suspended in the waste substance **(3)**.

3.1.15 *mixed waste glass*—a glass comprised of glass forming additives and both hazardous and radioactive constituents.

3.1.16 *multiphase glass ceramic waste form*—a ceramic consisting of more than one phase, one of which must be a glass.

3.1.17 *nuclear waste glass*—a glass comprised of glass forming additives and radioactive waste.

3.1.18 *open system tests*—a system that permits the transport of matter into or out of the system, for example, O₂ or CO₂ diffusion, or both, into or out of the system.

3.1.19 *phase separated glass*—a glass containing more than one amorphous phase.

3.1.20 *radioactive*—of or exhibiting radioactivity (*American Heritage Dictionary, 1973*); a material giving or capable of giving off radiant energy in the form of particles or rays, as alpha, beta, and gamma rays, by the disintegration of atomic nuclei; said of certain elements, such as radium, thorium, and uranium, and their products (*Webster's New Twentieth Century Dictionary, 1973*).

3.1.21 *radioactivity*—spontaneous nuclear disintegration with emission of corpuscular or electromagnetic radiation, or both (consult Terminology **D 1129**).

3.1.22 *sample blank*—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.

3.1.23 *sensitization*—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 **E 7**).

3.1.23.1 *Discussion*—This constitutes the greatest single threat to their corrosion resistance **(4)**.

3.1.24 *set of samples*—samples tested simultaneously in the same oven.

3.1.25 *simulated waste glass*—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.

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

3.1.27 *standardize*—to make, cause, adjust, or adapt to fit a standard **(3)**; to cause to conform to a given standard, for example, to make standard or uniform (*Webster's New Twentieth Century Dictionary, 1973*).

3.1.28 *unsensitized austenitic steel*—stainless steel that is not sensitized (see **sensitization**).

3.1.29 *verify*—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*)

3.1.30 *vitrification*—the process of fusing waste with glass making chemicals at elevated temperatures to form a waste glass (see Terminology **C 162**).

³ Varshneya, A. K., “Fundamentals of Inorganic Glasses,” Academic Press, Boston, MA (1994).

⁴ The boldface numbers in parentheses refer to the list of references at the end of these test methods.

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) (5). 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 reproducible, 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 U.S. Standard ASTM – 100 to + 200 mesh (0.149–0.074 mm), the particles are cleaned of adhering fines, and an 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 $10 \pm 0.5 \text{ cm}^3/\text{g}$ of sample mass (m_{solid})⁵ is added and the vessel is sealed. The vessel is placed in a constant temperature device at $90 \pm 2^\circ\text{C}$. The vessels must be placed in constant temperature devices so that there is ample convection around the samples and even heat distribution (Fig. 1). After seven days $\pm 3.4 \text{ h}$ the vessel is removed from the

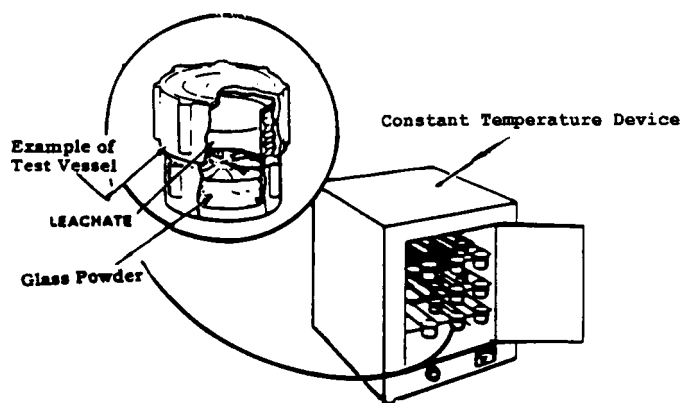


FIG. 1 Schematic of Test Apparatus

oven 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 sent for analysis.

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 (5). The test method is easily reproducible, 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 U.S. Standard ASTM – 100 to + 200 mesh (0.149–0.074 mm) or to 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 $10 \pm 0.5 \text{ cm}^3/\text{g}$ of sample mass (m_{solid})⁵ is added and the vessel is sealed. Other ratios of solution volume to sample mass are allowed and other leachants are allowed. The vessel is placed in a constant temperature device at $90 \pm 2^\circ\text{C}$. Other test temperatures are permissible. It is desirable that inter-comparison of test responses be conducted at different temperatures to indicate whether the reaction mechanism changes over the temperature range investigated. The vessels must be placed in a constant temperature device so that there is ample convection around the samples and even heat distribution (Fig. 1). After seven days $\pm 3.4 \text{ h}$, 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 sent for analysis.

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 3.1.4) of glass waste forms as measured by elemental release. Accordingly, it may be applicable throughout manufacturing, research, and development.

⁵ If waste forms of different densities are being compared then the leachate results from the test must be compared using the calculation in 25.2.4 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).

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 (6)	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 \pm 2%	7 days \pm 2% or varying times
Leachant	ASTM Type I water	ASTM Type I water or other solutions
Condition	Static	Static
Minimum Sample Mass	$\geq 1 \text{ g}$	$\geq 1 \text{ g}$
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	$10 \pm 0.5 \text{ cm}^3/\text{gram}$ of sample mass	$10 \pm 0.5 \text{ cm}^3/\text{gram}$ of sample mass or other volume/sample mass
Temperature	$90 \pm 2^\circ\text{C}$	$90 \pm 2^\circ\text{C}$ 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

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 leaching conditions, for example, varying test durations, test temperatures, ratio 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 [C 1174](#)).

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 to take internal pressures of >0.5 MPa without leaking (see Sections [10](#) and [11](#)).

6.1.1 The stainless steel vessels require a gasket material in order to remain sealed. 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 ([7](#)) not to damage TFE-fluorocarbon. If higher radiation doses are present, degradation of the TFE-fluorocarbon gasket can compromise the seal or contaminate the leachate with fluoride, or both, as F^- and HF ([8](#)). For high radiation doses, special gaskets fabricated from metals such as copper, gold, lead or indium are recommended. High radiation doses will not be experienced with hazardous, mixed, or simulated glass waste forms.

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 to take internal pressures of >0.5 MPa without leaking (see Section [10](#)) ([6](#)).

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

6.2.2 High radiation fields ($>1 \times 10^5$ rad of beta or gamma radiation) will not generally be generated by hazardous, mixed or simulated nuclear waste glass forms. PFA TFE-fluorocarbon vessels, available commercially, can be used in the absence of high radiation fields⁶ since PFA TFE-fluorocarbon is chemically inert when properly cleaned ([9](#)).

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

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

6.5 *Temperature Measurement Device*—Resistance thermometers or thermocouples, or both, with a strip chart recorder or a data logger for periodic monitoring of the temperature of the convection oven during the test duration. The maximum period between temperature recording frequency 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 mass 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 ([10](#)). 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 covers and receptacle, including the largest and smallest sieves for the desired size range.

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

6.11 *Pipets*—Calibrated pipets. Pipet tips that have either been precleaned, sterilized, or individually packaged to avoid contamination from handling.

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*—pH meter 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 [C 1109](#) or Ref [11](#), or both), atomic absorption spectrometry, ion chromatography (consult Test Method [D 4327](#) or Ref [11](#), or both), or ion selective electrodes, or a combination thereof.

7. Standards

7.1 *Reference Waste Form*—A reference waste form (see Ref [12](#) for example) of choice, similar in composition to the glass waste form being tested, is to be tested in triplicate along with each batch of glass waste forms tested (see Ref [13](#), [44](#)).

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.

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

The reference waste form composition should be traceable to NIST, or to a comparable source.

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 a comparable source and have a certified shelf life.

7.3 pH Buffers—commercial pH buffers or pH buffers made to the specifications given in Test Method D 1293 that bracket the measured pH range of the leachant and leachate. All commercial buffer solutions should be traceable to NIST, or a comparable source, 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 D 1293).

7.4 Analytic Standard Solutions—The reference solutions should be traceable to NIST, or a comparable source used. All standard solutions must have a certified shelf life.

8. Reagents and Standards

8.1 ACS Reagent Grade Acids—Reagent grade nitric acid (HNO₃) and hydrofluoric acid (HF) for cleaning leach vessels.⁸

8.2 ACS High Purity Acid—Ultra high purity concentrated nitric acid (HNO₃) 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 (95% pure) and 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 D 1193).

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/m³ (see Specification D 1193 and Terminology D 1129).

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 have a 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 55 mL, may lead to pressures inside the container that exceed the design limits and could lead to the failure of one or more parts of the vessel.

10. Choice of Test Vessel

10.1 Stainless Steel Vessels—Unsensitized Type 304L stainless steel vessels must be used in Test Method A and may be used in Test Method B. The user should ensure that the vessels are free from chloride (14). The user is also cautioned about the attraction of steel for certain radionuclides such as americium, plutonium, and other redox sensitive species.

10.1.1 Steel vessels represent “closed system” applications where the influx of CO₂ or O₂ into the leachate is not desired. The user is cautioned that the leachate concentrations and leachate pH in tests conducted in PFA TFE-fluorocarbon and steel vessels may be significantly different for some glass waste forms due to higher concentrations of CO₂ and O₂, in the “open system” tests performed in PFA TFE-fluorocarbon vessels, that is, the differences in “open” and “closed” system conditions. (15-23)

10.1.2 It is recommended that 22 mL vessels¹¹ be used for the radioactive production application in Test Method A 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. 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 ≤28 days. Longer test durations are also acceptable only if it can be demonstrated that the vessel interactions do not affect the glass waste form reactivity. The user should ensure that new PFA TFE-fluorocarbon vessels are free from fluoride which is present as a free surface fluoride from vessel fabrication (see Section 16).

10.2.1 PFA TFE-fluorocarbon vessels are for “open system” applications where the influx of CO₂ or O₂ into the leachant is either desirable or not of concern. The user is cautioned that the

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.

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.

leachate concentrations and leachate pH in PFA TFE-fluorocarbon and steel vessels may be different due to equilibration with CO₂ and O₂, that is, the differences in “open” and “closed” system conditions.

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 for radioactive glass waste forms⁶. The use of PFA TFE-fluorocarbon vessels for radiation doses >10⁵ rad 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 acidic conditions and F⁻ ions that attack the glass waste form (7, 8).

11. Identification of Vessels and Vessel Cleaning History

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

11.2 *Identification of Vessel Cleaning History*—Each batch of cleaned leach vessels will be labeled with a unique batch number. A log book of the leach 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 test 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 vessels. Degrease the vessels and lids in 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 95% absolute ethanol 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 HNO₃ (1 weight % HNO₃) and heat to 90 ± 10°C for a minimum of 1 h. The gasket should not be in the lid during the HNO₃ cleaning step because of the possibility that small amounts of HNO₃ may be trapped between the gasket and the lid.

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 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 [D 1293](#)).

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 and testing method starting at 12.1.4.

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 95% absolute ethanol 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 specimens, residual contamination may be present. The vessels shall be cleaned before reuse by cleaning with HNO₃ 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 0.16M HNO₃ (1 weight % HNO₃). Reseal the vessel and place in 90 ± 10°C oven for a minimum of 16 h to dissolve (acid strip) any 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 $90 \pm 10^\circ\text{C}$ 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 **D 1293**). 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 $>0.1 \text{ g/m}^3$ in the solution, 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 $>0.1 \text{ g/m}^3$ criteria cannot be achieved by three repetitions of 14.1.3 through 14.1.6, or a combination thereof, then repeat the cleaning and testing method starting at 14.1.1.

14.1.8 Dry vessels, lids, and gaskets at $90 \pm 10^\circ\text{C}$ 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 $90 \pm 10^\circ\text{C}$ 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 $90 \pm 10^\circ\text{C}$ 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 $90 \pm 10^\circ\text{C}$ 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 **D 1293**). 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 $>0.1 \text{ g/m}^3$ in the solution, repeat steps 15.1.4 through 15.1.7.

15.1.9 If the 5.0 to 7.0 pH range or the $>0.1 \text{ g/m}^3$ 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 $90 \pm 10^\circ\text{C}$ 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 (9). 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 $110 \pm 10^\circ\text{C}$ 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 to cool 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 $90 \pm 10^\circ\text{C}$ 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 **D 1293**).

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 $>0.5 \text{ }\mu\text{g/mL}$, 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 $90 \pm 10^\circ\text{C}$ 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 HNO₃ 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 HNO₃ (1 weight % HNO₃) 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 D 1293).

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 D 1293.

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 D 1125).

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 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 E 456 and E 1402). 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. 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 ($V_{\text{soln}}/m_{\text{solid}}$) is 10 ± 0.5 cm³/g. The volume of leachant is constrained by the volume of the test vessel chosen and the need to minimize sample size when dealing with highly radioactive glass waste forms. 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 shall be used to provide estimates of 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 and may alter the leachate results. Redox sensitive glass waste forms shall be used within two days of grinding (see Test Methods C 169 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 (10). Crushing devices made of Types 304L and 316L stainless steel should be used to minimize these effects. It is recommended that a representative amount 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 in order to have enough 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 (10). 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 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 C 225 and C 371 (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. A 200 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). Particle size analysis of samples sieved with more than 100 g of glass waste form on an 8 in. sieve has indicated a wider gaussian particle size distribution than for samples with <100 g of glass waste form on an 8 in. sieve.

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 device¹² shall be such as to 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 C 371). The mechanical sieving should continue for a minimum of 15 min (see Test Method C 429).

19.5.4.2 *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 C 92). Sieve for several minutes.

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

19.5.6 Tap the 200 mesh sieve forcefully over contrasting colored paper. For example, if the waste form is light colored tap the sieve on dark paper. For a dark colored waste form, for example, black nuclear waste glass, tap the sieve on white paper. Tap the sieve in an inclined position on one side; repeat several times tapping the inclined sieve on opposite sides each time. Continue the tapping until less than 0.1 g of the waste form passes through the sieve onto the paper after 1 min of continuous tapping (see Test Methods C 92). If >0.1 g of powder appears on the paper, repeat 19.5.4.1 through 19.5.6 until <0.1 g of powdered waste form passes through the 200 mesh sieve.

19.5.7 Transfer the – 100 to + 200 mesh fraction of the sieved glass waste form into a clean container labeled with the sample 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. When new waste form fragments have been prepared repeat 19.5.3 through 19.5.7.

19.5.9 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 and/or multiphase glass ceramic waste forms 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 crystalline or secondary amorphous phases on the test response. Secondary phases (crystalline or amorphous) present should be identified and either their potential dissolution rates relative to the bulk waste form considered, or the wash solutions analyzed directly. If preferential extraction is likely in the washing step, then 19.6.2 through 19.6.11 should be omitted from the test procedure and noted on the data sheet such as that given in Appendix X2. Alternatively, if the crystalline phases are known to be water soluble, steps 19.6.2 through 19.6.11 must be performed in fresh absolute ethanol. Any changes in the washing procedure 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 solvents.

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.

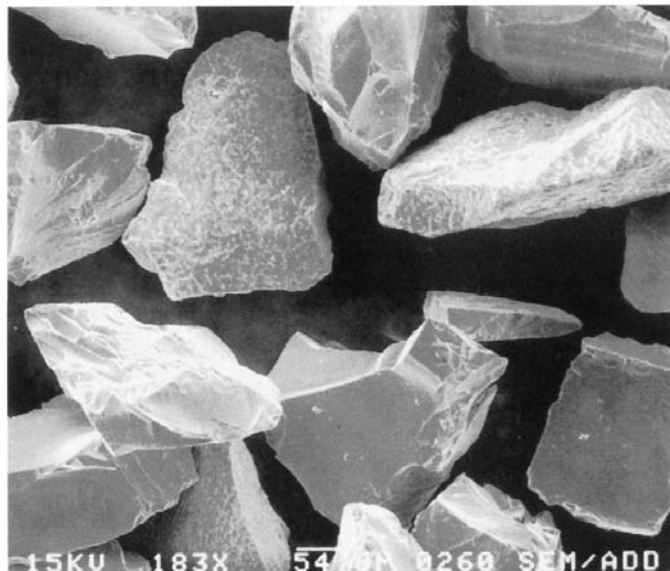


FIG. 2 Adherent fines on – 100 to + 200 mesh glass particles before washing (Top). Glass particles after washing in ASTM Type I Water and Absolute Ethanol (Bottom)

19.6.2 Place sieved waste form in a clean glass beaker that will hold about 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.

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

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 19.6.6 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.

19.6.10 Place the beaker from 19.6.9 in the ultrasonic cleaner for 2 min. After the 2 min decant the alcohol from the beaker.

19.6.11 Repeat 19.6.9 through 19.6.10 two more times.

19.6.12 Put the beaker containing the cleaned sample in a $90 \pm 10^\circ\text{C}$ 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 around while observing the sample under the microscope. If metal filings from the grinding are present they will stick to the magnetic tweezer making identification easier. If metal filings are present the sample must not be used. 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 dessicator or they have been stored in a dessicator for over three months they must be redried at $90 \pm 10^\circ\text{C}$ overnight. Drying the sample before weighing ensures that the powders do not contain adsorbed water when weighed and therefore ensures mass and surface area uniformity.

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.

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 “set” of unknown waste forms being tested. The same person should prepare the reference waste form using the same equipment that is used for the unknown waste forms being tested.

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—A standard glass waste form test 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 as the sample vessels but contains no waste form. For each set of samples, two blanks from the same batch of cleaned vessels shall be used. If more than one batch of cleaned vessels is used in a set of samples, then two blanks from each batch of vessels will be used.

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 electrical resistivity of 18 M Ω -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/m³ (see Specification [D 1193](#) and Terminology [D 1129](#)). 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 [D 1125](#)). 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 leach 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 ASTM Type I water equivalent to ten times the mass of glass waste form added as calculated in [21.4.5](#) so that ($V_{\text{soln}}/m_{\text{solid}} = 10 \pm 0.5$ 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 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 $90 \pm 2^\circ\text{C}$ oven. The seven day $\pm 2\%$ 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 $\pm 2\%$ 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 sample 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 leachate 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. Record the measured pH as the final test pH on a sample log sheet. Also record the temperature of the aliquot at the time 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. Initial all entries. Discard the aliquot used for the pH and temperature 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 ([5](#), [26](#)). 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 ([21.4.13](#)) 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 ([21.4.14](#)).

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](#).

[21.4.14](#) Acidify the leachate aliquots drawn for cation analysis with concentrated ultra high purity HNO₃ 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 HNO₃ acidification/dilution techniques can be used if necessary; the final diluted sample should contain at least 1% HNO₃ 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 C 1109 and D 4327](#)). The short-term precision of these analytic methods at concentrations at least 100 times the detection limit range are from 0.3 to 2% relative standard deviation. Precision degrades with decreasing concentration to approximately 25% relative standard deviation at approximately two times the detection limit. The detection limits for each analysis must accompany the reported results.

[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 powder, for example, visible changes in color, agglomeration, and physical characteristics. Wash the specimen powder from the leach container with pure water onto a clean watch glass and dry at $\leq 90 \pm 2^\circ\text{C}$. A temperature of $90 \pm 2^\circ\text{C}$ will only drive off adsorbed moisture and not water of hydration. After drying, store in a clean container or analyze (e.g., consult [Practice C 1463](#)).

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.

[22.2 Choice of Appropriate Sample](#)—Samples of glass waste forms may either be fabricated individually or taken from larger samples (see [Terminologies E 456 and E 1402](#)). 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. 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 g. The recommended ratio of leachant volume to sample mass ($V_{\text{soln}}/m_{\text{solid}}$) is 10 ± 0.1 mL/g. Other $V_{\text{soln}}/m_{\text{solid}}$ can be used. The volume of leachant is constrained by the volume of the test vessel chosen.

[22.4 Number of Sample Replicates](#)—A minimum of three replicate samples shall be used to provide estimates of experimental variability.

[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 and may alter the leachate results. Redox sensitive glass waste forms shall be used within two days of grinding (see [Test Methods C 169 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 ([10](#)). 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.

[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 ([10](#)). 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 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 [C 225](#) and [C 371](#)). (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](#)). Particle size analysis of samples sieved with more than 100 g of glass waste form on an 8 in. sieve has indicated a wider Gaussian particle size distribution than for samples with <100 g of glass waste form on an 8 in. sieve.

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 device¹² shall be such as to 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 [C 371](#)). The mechanical sieving should continue for a minimum of 15 min (see Test Method [C 429](#)).

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 [C 92](#)). 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 less than 0.1 g of the waste form passes through the sieve onto the paper after 1 min of continuous tapping (see Test Methods [C 92](#)). If >0.1 g of powder appears on the paper, repeat [22.5.4](#) through [22.5.6](#) until <0.1 g of powdered waste form passes through the smallest mesh sieve.

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 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 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. 2](#)). However, devitrified, phase separated, and/or multiphase glass ceramic waste forms 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 crystalline or secondary amorphous phases on the test response. Secondary phases present should be identified and either their potential dissolution rates relative to the bulk waste form considered, or the wash solutions analyzed directly. If preferential extraction is likely in the washing step, then [22.6.2](#) through [22.6.11](#) should be omitted from the test procedure and noted on the data sheet such as that given in [Appendix X2](#). Alternatively, if the crystalline phases are known to be water soluble, steps [22.6.2](#)–[22.6.11](#) can be performed in fresh absolute ethanol. Any changes in the washing procedure 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 radioactive EPA hazardous solvents.

22.6.2 Place sieved glass waste form in a clean glass beaker that will hold about 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.

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 [22.6.6](#) and [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.

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.

22.6.11 Repeat [22.6.9](#) through [22.6.10](#) two more times.

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 around while observing the glass under the microscope. If metal filings from the grinding are present they will stick to the magnetic tweezer making identification easier. If metal filings are present the sample must not be used. 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 over three months they must be redried at $90 \pm 10^\circ\text{C}$ overnight. Drying the sample before weighing ensures that the powders do not contain adsorbed water when weighed and therefore ensures mass and surface area uniformity.

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.

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 “set” of unknown glass waste forms being tested. The same sample mesh size, $V_{\text{soln}}/m_{\text{solid}}$, 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.

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*—A standard glass waste form test 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 cleaned test vessel that has been filled with the same amount of ASTM Type I water as the sample vessels but contains no waste form. For each set of samples, two blanks from the same batch of cleaned vessels shall be used. If more than one batch of cleaned vessels is used in a set of samples, then two blanks from each batch of vessels will be used.

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 $18 \text{ M}\Omega\cdot\text{cm}$ 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 D 1193 and Terminology D 1129). 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 D 1125). 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 leach 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 the appropriate amount of leachant equivalent to the $V_{\text{soln}}/m_{\text{solid}}$ chosen. A $V_{\text{soln}}/m_{\text{solid}}$ of $10 \pm 0.5 \text{ mL/g}$ 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 but no 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 $90 \pm 2^\circ\text{C}$. The desired test period starts at this time. The test duration equivalent to PCT-A is seven days $\pm 2\%$. 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 $\pm 2\%$ 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 sample 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 leachate 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. Record the measured pH as the final test pH on a sample log sheet. Also record the temperature of the aliquot at the time 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. 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 (5, 26). 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 (24.4.13) 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 (24.4.14).

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.

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 C 1109 and D 4327). The short-term precision of these analytic methods at concentrations at least 100 times the detection limit range are from 0.3 to 2% relative standard deviation. Precision degrades with decreasing concentration to approximately 25% relative standard deviation at approximately two times the detection limit. The detection limits for each analysis must accompany the reported results.

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 powder, for example, visible changes in color, agglomeration, and physical characteristics. Wash the specimen powder from the leach container with pure water onto a clean watch glass and dry at $\leq 90 \pm 2^\circ\text{C}$. A temperature of $90 \pm 2^\circ\text{C}$ will only drive off adsorbed moisture and not water of hydration. After drying, store in a clean container or analyze (e.g., consult Practices C 1463).

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 of the study 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 themselves (probably due to improper cleaning) or are present in the original leachant itself (whether leachant is ASTM Type I water or a groundwater). A blank element concentration is considered significant if it is $>10\%$ of the concentration of that respective element in the leachate when the glass waste form was present in the test. Leachate concentrations, especially those for the major soluble elements in the glass waste form (see Section 26) are measured for the replicate blanks. The mean concentration of the replicate blanks is used to correct all the sample leachate

analyses. If corrections of >10% of any element occur, data for that element are invalid.

25.2 Calculations of Leachate Concentrations—Leachate concentrations, especially those for the major soluble elements in the glass (see Section 26) are calculated for the waste form being tested and the standard waste form. Standard waste form leachate concentrations and pH values should be control charted. Concentrations from each new set of standard analyses should be compared to previous standard test responses. If the current standard data is within the control chart range then all of the leachate test data can be compared to previous data sets. If the data is outside the normal control chart range for this standard, then the data for all the samples must be bias corrected before it can be used. 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 intra-laboratory variables (13).

25.2.1 Calculate the final leachate volume for each test including blanks. The amount of water loss is equal to the weight loss that occurred during heating. Leachate losses of greater than 5% of the initial volume invalidate Test Method A data, while leachate losses of greater than 5% of the initial volume invalidate Test Method B data (see 21.4.9 and 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:

$$C_i(\text{blank}) = \left(\sum_1^m C_i(m) \cdot \frac{V_f(m)}{V_i(m)} \right) \cdot \frac{1}{m}$$

where:

$C_i(\text{blank})$ = average blank concentration, g/L,
 $C_i(m)$ = concentration observed in the m^{th} blank, g/L,
 $V_i(m)$ = initial volume of leachate blank, L,
 $V_f(m)$ = final volume of the leachate blank, L, and
 m = number of replicate blank tests performed.

25.2.3 Calculate the corrected leachate concentration for each sample replicate by subtracting the average blank concentration from the measured concentration. Note that for a valid test, the blank concentrations for the elements of concern are to be <10% of the leachate concentrations. If the blank concentrations for a given element are greater than three times the detection limit of the analytic method used for all replicate blanks, then the equation below 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 of the analytic method used for all replicate blanks, for example, in the range where the analysis may be in error by 100 to 200%, 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) in only one replicate blank, then all vessels from that cleaning batch need to be reevaluated for elevated concentrations of that element since this may be an indication that some of the vessels may not have been adequately cleaned. A

decision must be made as whether to blank correct using the equation below or not.

The following equation can be used to blank correct each replicate analysis:

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

where:

$C_i(n)$ = concentration observed in the n^{th} waste form sample, g/L,
 $V_f(n)$ = final volume of leachate measured in the n^{th} waste form sample replicate, L,
 $V_i(n)$ = initial volume of leachate in the n^{th} waste form sample replicate, L,
 n = total number of replicate waste form sample tests performed, and
 $C_i(\text{blank})$ = average blank concentration, g/L.

25.2.4 Calculating the concentration in normalized mass units NC_i or $\log[NC_i]$ is recommended for all waste forms including multiphase waste forms because the release concentrations in g/L are normalized by the weight fraction of that element present in the waste form. The normalized release, NC_i , is a function of (1) the mass fraction of the i^{th} element of interest in the glass and (2) the concentration of the i^{th} species in solution.¹³ The normalized concentration for each replicate is expressed as:

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

where:

NC_i = normalized concentration, $\text{g}_{\text{waste form}}/\text{L}_{\text{leachant}}$,
 $c_i(\text{sample})$ = concentration of element “ i ” in the solution, g/L, and
 f_i = fraction of element “ i ” in the unleached waste form (unitless).

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}), for example, $V_{\text{soln}}/m_{\text{solid}} = 10 \pm 0.5 \text{ cm}^3/\text{g}$, and at 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 leachant (V) at a constant. As long as the glass waste form density and glass waste form particle size remain comparable between leach tests, this parameter will remain a constant and need not be calculated every time. Since triplicate PCT analyses must be performed on a glass waste form and composition is normally based on one or more analyses, there is not a one-to-one correspondence between the uncertainties in the solution (C_i) and glass (f_i) concentrations. Thus the appropriate manner in which to compute, and thus report, the release term in order to minimize uncertainty is:

$$\log(NC_i) = \overline{\log(C_i)} - \overline{\log(f_i)}$$

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

where:

$\overline{\log(Ci)}$ = average of the logarithms of the corrected solution concentrations, and

$\overline{\log(fi)}$ = average of the logarithm of the glass concentrations.

Alternatively, a normalized release rate, NR_i , can be calculated when the particle size, $V_{\text{solid}}/m_{\text{solid}}$, and time are varied between tests, or for test results on waste forms of different density, the following expression may be used to calculate NR_i :

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

where:

NR_i = normalized rate, $g_{\text{waste form}}/m^2 \cdot \text{day}$,

$c_i(\text{sample})$ = concentration of element “i” in the solution, g_i/L ,

f_i = fraction of element “i” in the unleached waste form (unitless),

SA/V = surface area of the final waste form divided by the leachate volume, m^2/L , and

t = time duration of test in days.

The units of NR_i for PCT-B may normally expressed as grams of glass waste form dissolved per $m^2 \cdot \text{day}$. If the units of time are omitted from the NR_i calculation, then the normalized release, NL_i , is calculated as:

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

where:

NL_i = normalized release, $g_{\text{waste form}}/m^2$,

$c_i(\text{sample})$ = concentration of element “i” in the solution, g_i/L ,

f_i = fraction of element “i” in the unleached waste form (unitless), and

SA/V = surface area of the final waste form divided by the leachate volume, m^2/L .

25.2.5 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 represent the maximum dissolution of the glass waste form. For example, elements that are not sequestered in precipitates 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 (27, 32-42) of any glass or glass ceramic waste form must be performed in order to determine what these elements are. Often the maximum normalized release (see Section 25) of the radioactive constituents can be compared to the release of non-radioactive indicator(s) elements that can be more readily and accurately measured. The nonradioactive indicator(s) should be capable of representing radionuclides that are present at concentrations as low as 10^{-8} weight %, and/or radionuclides, that are difficult to measure. Once the pertinent indicator(s) is(are) determined for a given glass waste form, these

constituents should be analyzed in the leachate. For example, in high level borosilicate waste glass, Tc⁹⁹, present at $\sim 4.1 \times 10^{-4}$ weight % in the waste form, has been shown to be released at the same maximum normalized concentration as boron, lithium, and sodium. 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 at > 1 weight % 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 Practice C 1463).

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 Bias¹⁴

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

NOTE 10—Precision cited from Refs 5 and 26 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 (25).

27.1.1 The data used to generate the measures of precision for PCT Test Method B in TFE-fluorocarbon are the result of intra- and interlaboratory round robins (5, 26). These measures are typical of the methods as applied to the glasses and standards used in the round robins, and are not all inclusive with respect to other types of glasses. The measures of precision were determined in accordance with procedures in Practices E 177 and E 691. 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 has recently been 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 (43) (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 (ppm) of silicon and boron in the PCT leachates was established using washed -100 to +200 mesh glass in ASTM Type I water for

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

seven days (data in Ref [43](#), Table 6). Each laboratory performed triplicate tests on the LRM glass. For silicon the relative within laboratory precision was 4.3%. For boron, the value was 6.7%.

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 [27](#), Table 3). 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 [27](#), Table 4). 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 ([12](#)), 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 [28](#), Table 2). 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 has recently been 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 ([43](#)) ([27.1.3.1](#)). Data from two separate laboratories have been used to determine the between laboratory precision for remote radioactive operation ([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 (ppm) of silicon 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 [43](#), Table 6). Each laboratory performed triplicate tests on the LRM glass. For silicon, the relative between laboratory precision was 15.4%. For boron, the value was 27.0%.

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 [27](#), Table 3). 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 activa-

tion 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 [27](#), Table 4). Each laboratory performed triplicate tests on each glass.

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

27.1.4.1 Approved Reference Material (ARM-1, a simulated borosilicate nuclear waste glass ([12](#)))—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 ([5](#)). 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 sodalite and other minor ceramic phases)—A round robin was conducted with four participating laboratories in TFE-fluorocarbon vessels at the reference PCT-A conditions. Two solutions were measured during the round robin, the water wash solution ([19.6.1](#)) and the primary test solution ([21.4.13](#)). The within-laboratory relative standard for boron and lithium from the borosilicate glass was 8.1% and 5.3% respectively. Sodium from the borosilicate glass and the sodalite and other minor sodium containing phases had a %RSD of 13.4% in the test solutions. Likewise, silicon which participates in the glass and in the ceramic sodalite phase had a %RSD of 2.6% in the test solution. The %RSD for chloride which is present in the ceramic phase sodalite and an NaCl phase was 8.2% in the test solution.

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 ([12](#)))—Between-laboratory 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 [29](#), Table C.2). 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 [29](#), Table C.2). 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 29, Table C.2). 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 sodalite and other minor ceramic phases)—A round robin was conducted with four participating laboratories in TFE-fluorocarbon vessels at the reference PCT-A conditions. Two solutions were measured during the round robin, the water wash solution (19.6.1) and the primary test solution (21.4.13). The between-laboratory relative standard deviation (including

within laboratory and between laboratory %RSD) for boron and lithium from the borosilicate glass was 19% and 12% respectively. Sodium from the borosilicate glass and the sodalite and other minor sodium containing phases had a %RSD of 18% in the test solutions and 7% in the wash solution. Likewise, silicon which participates in the glass and in the ceramic sodalite phase had an %RSD of 20% in the test solution. The %RSD for chloride which is present in the ceramic phase sodalite and an NaCl phase was 26% in the test solution and 6% in the wash solution.

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 (13).

APPENDIXES

(Nonmandatory Information)

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

X1.1 Glass waste form particles are neither spherical, tabular, nor square (see Fig. 2). In order to calculate exposed glass waste form surface area from the mass of glass waste form used, one must assume a given geometric shape. If the glass waste form is assumed to be spherical and the particle size distribution between the – 100 (0.149 mm) to + 200 (0.074 mm) mesh is assumed to be Gaussian the following

calculation allows an estimated glass surface area to be calculated. Particle size analyses of well sieved samples (see Fig. 3) shows that use of the average diameter of the –100 to +200 mesh particles (1.12×10^{-4} m) introduces no significant error. Particle size analysis was examined for both hand sieving and mechanical sieving (for example, consult Test Method C 429) and shown to be Gaussian as shown in Fig. 3. For this

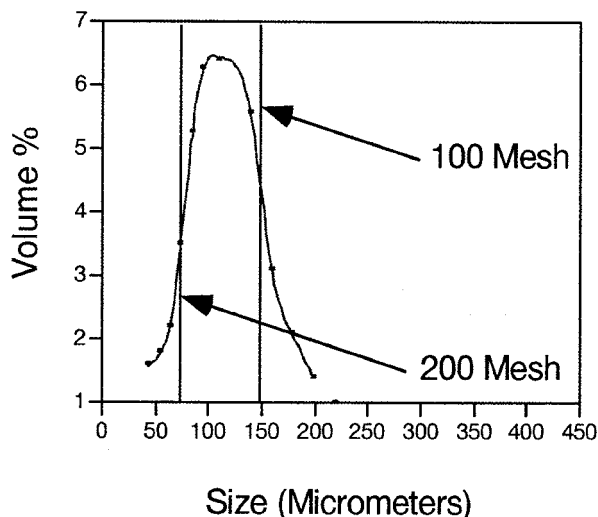


FIG. 3 Example of the Particle Size Distribution on the –100 to +200 Mesh Sieve

example the glass waste form density (for example, consult Test Method **C 693**) is assumed to be 2.7 g/cc and it is assumed that the researcher has used a m_{solid} to volume ratio of 4 g of glass to 40 cc of water. The area (A) and volume (V) of a spherical particle are given by

$$A = \Pi d^2 \quad \text{and} \quad V = (1/6) \Pi d^3$$

where d is the average particle diameter, 1.12×10^{-4} m for – 100 + 200 mesh and Π is 3.14. The area and volume of an average spherical – 100 + 200 mesh size particle is, therefore, 3.90×10^{-8} m² and 7.25×10^{-13} m³, respectively. Since the assumed glass density is 2.7 g/cc or 2.7×10^6 g/m³ and density is mass/unit volume, the mass of an average size particle can be determined as

$$\text{average particle mass} = \text{density} * (\text{volume})$$

$$\text{average particle mass} = (2.7 \times 10^{-6} \text{ g/m}^3) (7.25 \times 10^{-13} \text{ m}^3) = 1.96 \times 10^{-6} \text{ g}$$

This enables the calculation of the number of particles per gram of crushed glass waste form, for example, there are 5.11×10^5 particles in 1 g of – 100 to + 200 mesh glass waste form, for example, $1 \text{ g} / 1.96 \times 10^{-6} = 5.11 \times 10^5$ particles. Each particle has a surface area of 3.90×10^{-8} m² as given above. The total calculated surface area in 1 g of – 100 to + 200 mesh glass (A per gram) is A per gram = $(5.11 \times 10^5 \text{ particles}) (3.90 \times 10^{-8} \text{ m}^2/\text{particle}) = 1.99 \times 10^{-2} \text{ m}^2$. The calculated surface area in 4 g is $8 \times 10^{-2} \text{ m}^2$. As long as the glass waste form density and glass waste form particle size remain comparable between leach tests, this parameter will remain a constant and need not be calculated every time. Assuming cubic (**30**) or tabular (**31**) shapes imparts only an approximately 1 % difference in the surface area calculation compared to assuming spherical shapes.

NOTE X1.1—For tabular shapes the error could be greater depending on the aspect ratio of the particles.

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. 1 st Vessel Blank	ID#	Batch cleaning #			
3. 2 nd 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 meter 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:

Acidified 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:

REFERENCES

- (1) Federal Register 52FR, Department of Energy, 10CFR Part 962, Final Rule, May 1, 1987, p. 15 937.
- (2) Environmental Protection Agency Federal Regulation 40 CFR 261.
- (3) Department of Energy Order 5400.3
- (4) Zapffe, C. A., "Stainless Steels," The American Society for Metals, Cleveland, OH, 1949.
- (5) "Product Consistency Test (PCT) for DWPF Glass, Part I: Test Development and Protocol," *U.S. Department of Energy Report DPST-87-575*, Savannah River Laboratory, Aiken, SC, July 30, 1987.
- (6) *U.S. Department of Energy Report EM-WAPS*, Waste Acceptance Product Specifications for Vitrified High-Level Waste Forms, Revision 2, December 1996.
- (7) Strachan, D. M., "Effect of Gamma Irradiation on Simulated Waste Glass Leaching and on the Leach Vessel," *Journal of the American Chemical Society*, Vol 66, Number 9, C-158-C-160, 1983.
- (8) Bibler, N. E., "Leaching Fully Radioactive SRP Nuclear Waste Glass in Tuff Groundwater in Stainless Steel Vessels," Nuclear Waste Management, II, Clark, D. E. et al, eds., *Advances in Ceramics*, Vol 20, American Ceramic Society, Westerville, OH, 1986, pp. 619–626.
- (9) Goodman, J., and Mudrak, L., "Chemical Carryover from PFA Process Wafer Carriers, Solid State Technology," *Introduction to Microwave Sample Preparation, Theory and Practice*, Kingston, H. M., and Jassie, L. B., eds., American Chemical Society, Washington, DC, 1988, p. 263.
- (10) McVay, G. L., and Buckwalter, C. Q., "Effect of Iron on Waste Glass Leaching," *Journal of the American Ceramic Society*, Vol 66, 1983, pp. 170–174.
- (11) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846A (latest version or equivalent)," *SW846A*, 3rd edition, Revision 1, U.S. Environmental Protection Agency, Washington DC, December 1987.
- (12) Mellinger, G. B., and Daniel, J. L., "Approved Reference Materials for Use in Nuclear Waste Management Research and Development Programs," *Department of Energy Report PNL-4955-2*, Pacific Northwest Laboratories, Richland, WA, December 1984; NIST Standard Reference Material SRM-623. Smith, G. L., "Characterization of Analytical Reference Glass-1 (ARG-1)," Technical Report PNL-8992 Pacific Northwest National Laboratory, Richland, WA, December 1993.
- (13) *ASTM Manual on Presentation of Data and Control Chart Analysis*, Special Technical Publication 15D.
- (14) Gray, W. J., "Effects of Radiation on the Oxidation Potential of Salt Brine, Scientific Basis for Nuclear Waste Management, XI," *Materials Research Society Symposium*, Volume 112, 1987, p. 405.
- (15) Jantzen, C.M., Bibler, N.E., Beam, D.C. and Pickett, M.A., "Development and Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material." Environmental and Waste Management Issues in the Ceramic Industry, *Ceramic Transactions*, 39, American Ceramic Society, Westerville, OH, 313-322 (1994).
- (16) Jantzen, C.M., Bibler, N.E., Beam, D.C., Crawford, C.L. and Pickett, M.A. "Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material," U.S. DOE Report WSRC-TR-92-346, Rev. 1, 92p (June, 1993).
- (17) Jantzen, C.M., Bibler, N.E., Beam, D.C., Crawford, C.L., Marra, S.L., Ramsey, A.A., and Pickett, M.A. "Development and Characterization of the Defense Waste Processing Facility (DWPF) Waste Compliance Plan (WCP) Glasses," U.S. DOE Report WSRC-TR-93-181 (in preparation).
- (18) Park, P.K., "Oceanic CO₂ System: An Evaluation of Ten Methods of Investigation," *Limnology and Oceanography*, 14121, 179-186 (1969).
- (19) Whitefield, M. And Jagner D., *Marine Electrochemistry. A Practical Introduction*, John Wiley and Sons, 529pp - see Chapter 1, (1981).
- (20) Knauss, K.G., Nguyen, S.N., and Weed, H.C. "Diopside Dissolution Kinetics as a Function of pH, CO₂, Temperature, and Time," *Geochimica et Cosmochimica Acta*, 57, 285-294 (1993).
- (21) Xing, S.B. and Pegg, I.L., "Effects of Container Material on PCT Leach Test Results for High-Level Nuclear Waste Glasses," *Scientific Basis for Nuclear Waste Management, XVII*, A. Barkatt and R.A. VanKonynenburg (eds.), Materials Research Society, Pittsburgh, PA 557-564 (1994).
- (22) Olson, K.M. and Smith, G.L., "Product Consistency Testing of Three Reference Glasses in Stainless Steel and Teflon Vessels," *Environmental Issues and Waste Management Technologies in the Ceramic and Nuclear Industries, Ceramic Transactions*, 61, The American Ceramic Society, Westerville, OH, 523-530 (1995).
- (23) Olson, K.M., Smith, G.L., and Marschman, S.C., "Product Consistency Testing of Three Reference Glasses in Stainless Steel and Perfluoroalkoxy Resin Vessels," Technical Report PNL-10497, Pacific Northwest National Laboratory, Richland, WA, (March, 1995).
- (24) Torres Sanchez, R. M., Curt, E. M., Volzone, C., Mercader, R. C., and Cavalieri, A. L., "Study of Fe(II) Oxidation in Ground Magnetite," *Materials Research Bulletin*, Vol 25, 1990, pp. 553–561.
- (25) Jantzen, C. M., "Evaluation of Experimental Factors that Influence the Application and Discrimination Capability of the Product Consistency Test," *U.S. Department of Energy Report WSRC-TR-90-526*, Westinghouse Savannah River Co., October 1990.
- (26) Vernaz, E., Advocat, T., and Dussosoy, J. L., "Effects of the SA/V Ratio on the Long-Term Corrosion Kinetics of R7-T7 Glass," Nuclear Waste Management, III, Mellinger, G. B., ed., *Ceramic Transactions*, Vol 9, American Ceramic Society, Westerville, OH, 1990, pp. 175–185.
- (27) Bibler, N. E., and Bates, J. K., "Product Consistency Leach Tests of Savannah River Site Radioactive Waste Glasses," *Scientific Basis for Nuclear Waste Management, XIII*, Oversby, V. M. and Brown, P. W., eds., Materials Research Society, Pittsburgh, PA, 1990, pp. 327–338.
- (28) Manaktala, H. K., "Leaching of Borosilicate Glass Using Draft ASTM Procedure for High-Level Waste," *U.S. Report CNWRA 92-018*, Center for Nuclear Waste Regulatory Analyses, San Antonio, TX, August 1992.
- (29) Piepels, G. F., Jones, P. E., Eggett, D. L., and Mellinger, G. B. "Product Consistency Test Round Robin Conducted by the Materials Characterization Center—Summary Report," U.S. Department of

- Energy Report PNL-6967, Battelle Pacific Northwest Laboratory, Richland, WA, September 1989.
- (30) Shade, J. W. and Strachan, D. M., "Effect of High Surface Area to Solution Volume Ratios on Waste Glass Leaching," *American Ceramic Society Bulletin*, Vol 65, Issue 12, 1986, pp. 1568–1573.
- (31) Pine, G. L. and Jantzen, C. M., "Implications of a One-Year Basalt Weathering/Reactivity Study for a Basalt Repository Environment," *U.S. DOE Report DP-1742*, E. I. duPont deNemours and Co., Savannah River Laboratory, Aiken, SC, March 1987.
- (32) Bates, J. K., Lam, D. J., and Steindler, M. J., "Extended Leach Studies of Actinide-Doped SRL 131 Glass," *Scientific Basis for Nuclear Waste Management*, VI, D. G. Brookins (ed.), North-Holland, NY, 1983, pp. 183–190.
- (33) Bibler, N. E. and Jurgensen, A. R., "Leaching Tc-99 from SRP Glass in Simulated Tuff and Salt Groundwaters," *Scientific Basis for Nuclear Waste Management*, XI, M.J. Apted and R.E. Westerman (eds.), Materials Research Society, Pittsburgh, PA, 1988, pp. 585–593.
- (34) Bradley, D. J., Harvey, C. O., and Turcotte, R. P., "Leaching of Actinides and Technetium from Simulated High-Level Waste Glass," *Pacific Northwest Laboratory Report*, PNL-3152, Richland, WA, 1979.
- (35) Fillet, S., Nogues, J., Vernaz, E., and Jacquet-Francillon, N., "Leaching of Actinides from the French LWR Reference Glass," *Scientific Basis for Nuclear Waste Management*, IX, L. O. Werme, Materials Research Society, Pittsburgh, PA, 1985, pp. 211–218.
- (36) Bazan, F., Rego, J., and Aines, R. D., "Leaching of Actinide-doped Nuclear Waste Glass in a Tuff-Dominated System," *Scientific Basis for Nuclear Waste Management*, X, J. K. Bates and W. B. Seefeldt (eds.), Materials Research Society, Pittsburgh, PA, 1987, pp. 447–458.
- (37) Vernaz E. Y. and Godon, N., "Leaching of Actinides from Nuclear Waste Glass: French Experience," *Scientific Basis for Nuclear Waste Management*, XV, C. G. Sombret (ed.), Materials Research Society, Pittsburgh, PA, 1992, pp. 37–48.
- (38) Ebert, W. L., Wolf, S. F., and Bates, J. K., "The Release of Technetium from Defense Waste Processing Facility Glasses," *Scientific Basis for Nuclear Waste Management*, XIX, W. M. Murphy and D. A. Knecht (eds.), Materials Research Society, Pittsburgh, PA, 1996, pp. 221–227.
- (39) McGrail, B. P., "Waste Package Component Interactions with Savannah River Defense Waste Glass in a Low-Magnesium Salt Brine," *Nuclear Technology*, 1986, pp. 168–186.
- (40) Morss, L. R., Stanley, M. L., Tatko, C. D., and Ebert, W. L., "Corrosion of Glass Bonded Sodalite as a Function of pH and Temperature," *Scientific Basis for Nuclear Waste Management*, XXIII, R. W. Smith and D. W. Shoesmith (eds.), Materials Research Society, Pittsburgh, PA, 2000, pp. 733–738.
- (41) Sinkler, W., O'Holleran, T. P., Frank, S. M., Richmann, M. K., and Johnson, S. G., "Characterization of A Glass-Bonded Ceramic Waste Form Loaded with U and Pu," *Scientific Basis for Nuclear Waste Management*, XXIII, R. W. Smith and D. W. Shoesmith (eds.), Materials Research Society, Pittsburgh, PA, 2000, pp. 423–429.
- (42) Moschetti, T., Sinkler, W., Disanto, T., Hois, M. H., Warren, A. R., Cummings, D., Johnson, S. G., Goff, K. M., Bateman, K. J., and Frank, S. M., "Characterization of a Ceramic Waste Form Encapsulating Radioactive Electrorefiner Salt," *Scientific Basis for Nuclear Waste Management*, XXIII, R. W. Smith and D. W. Shoesmith (eds.), Materials Research Society, Pittsburgh, PA, 2000, pp. 577–582.
- (43) Ebert, W. L. and Wolf, S. F., "An Intralaboratory Study of a Standard Glass for Acceptance Testing of Low-Activity Waste Glass," *Journal of Nuclear Materials*, Vol 282, 2000, pp. 112–124.
- (44) Ebert, W. L. and Wolf, S. F., "Round Robin Testing of a Reference Glass for Low-Activity Waste Forms," Department of Energy Report ANL-99/22, Argonne National Laboratory, Argonne, Illinois, 1999.

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