**Designation: C 1285 – 97**

# Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses: 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 and devitrified glasses by measuring the concentrations of the chemical species released from a crushed glass to a test solution.

1.1.1 Test Method A is a seven-day crushed glass chemical durability test performed at 90 ± 2°C in a leachant of ASTM-Type I water. The test method is static and conducted in stainless steel vessels. Test Method A can specifically be used to evaluate whether the chemical durability and elemental release characteristics of nuclear, hazardous, and mixed waste glasses have been consistently controlled during production. This test method is applicable to radioactive and simulated waste glasses.

1.1.2 Test Method B is a crushed glass durability test that allows testing of waste glasses at varying test durations, test temperatures, mesh size, mass of glass, leachant volume, and leachant types. 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 or devitrified glasses, or both. This test method is applicable to radioactive (nuclear) and mixed, hazardous, and simulated waste glasses. Test Method B cannot be used as a consistency test for production of high level radioactive waste glass.

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

1.3 *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:*

C 92 Test Methods for Sieve Analysis and Water Content of Refractory Materials[2]

C 162 Terminology of Glass and Glass Products[3]

C 169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass[3]

C 225 Test Methods for Resistance of Glass Containers to Chemical Attack[3]

C 371 Test Method for Wire-Cloth Sieve Analysis of Nonplastic Ceramic Powders[3]

C 429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture[3]

C 693 Test Method for Density of Glass by Buoyancy

C 1109 Test Method for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectrometry[4]

C 1174 Practice for Prediction of the Long-Term Behavior of Materials, Including Waste Forms, Used in Engineered Barrier Systems (EBS) for Geologic Disposal of High–Level Radioactive Waste[4]

C 1317 Practice for Dissolution of Silicate or Acid Resistant Matrix Samples[4]

C 1342 Practice for Flux Fusion Sample Dissolution[4]

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water[5]

D 1129 Terminology Relating to Water[5]

D 1193 Specification for Reagent Water[5]

D 1293 Test Methods for pH of Water[5]

D 4327 Test Method for Anions in Water by Chemically Suppressed Ion Chromatography[5]

E 7 Terminology Relating to Metallography[6]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[7]

E 456 Terminology Relating to Quality and Statistics[7]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[7]

E 1402 Terminology Relating to Sampling[7]

## 3. Terminology

3.1 *Definitions:*

3.1.1 *anneal*—to prevent or remove materials processing

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[1] These test methods are under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.13 on Repository Waste.

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[2] *Annual Book of ASTM Standards*, Vol 15.01.

[3] *Annual Book of ASTM Standards*, Vol 15.02.

[4] *Annual Book of ASTM Standards*, Vol 12.01.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

[6] *Annual Book of ASTM Standards*, Vol 03.01.

[7] *Annual Book of ASTM Standards*, Vol 14.02.

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/m$^3$, a maximum electrical conductivity of 0.056 μmho/cm at 25°C, a minimum electrical resistivity of 18 MΩ·cm at 25°C (see Specification D 1193 and Terminology D 1129).

3.1.4 *chemical durability— in these test methods*, the resistance of a glass test specimen 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 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*—glass that has crystallized during cooling or due to thermal heat treatment, or both.

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

3.1.9 *glass*—an inorganic product of fusion that has cooled to a rigid condition without crystallizing (see Terminology C 162).

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

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

3.1.12 *mixed waste*—waste containing both radioactive and hazardous components regulated by the Atomic Energy Act (AEA) **(1)**[8] 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)**.

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

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

3.1.15 *open system tests*—a system that permits the transport of matter into or out of the system, for example, $O_2$ or $CO_2$ diffusion, or both, into or out of the system.

3.1.16 *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*).

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

3.1.18 *sample blank*—a cleaned test vessel that has been filled with the same amount of leachant as the sample vessels but contains no glass sample.

3.1.19 *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).

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

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

3.1.21 *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.

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

3.1.23 *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)*.

3.1.24 *unsensitized austenitic steel*—stainless steel which is not sensitized (see **sensitization**).

3.1.25 *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*)

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

## 4. Summary of Test Methods

4.1 Test Method A is the Product Consistency Test (PCT-A), which was developed specifically to test the chemical durability of radioactive waste glasses during production (Table 1) **(5)**. It can also be used to test hazardous, mixed, and various simulated waste glasses. The test method is easily reproducible, can be performed remotely on highly radioactive samples and can yield results rapidly. The glass does not need to be annealed prior to testing. In this test method the glass 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 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 cc/g of sample mass ($m_{solid}$) is added and the vessel is sealed. The vessel is placed in a constant temperature device at 90 ± 2°C. The vessels must be placed in the constant temperature devices so that there is ample convection around the samples and even heat distribution (Fig. 1). After seven days ±3.4 h the vessel is removed from the 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 test the chemical durability of radioactive, mixed, or simulated waste glasses **(5)**. The test method

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

TABLE 1 Summary of Test Methods A and B

| | Test Method A | Test Method B |
|---|---|---|
| Type of Glass | Radioactive<br>Mixed<br>Simulated, Hazardous | Radioactive<br>Mixed<br>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 9) |
| Test Duration | 7 days ± 3.4 h | 7 days ± 3.4 h or varying times |
| Leachant | ASTM Type I water | ASTM Type I water or other solutions |
| Condition | Static | Static |
| Sample Mass | ≥1 g | ≥1 g |
| Particle Size | U.S. Standard ASTM − 100 to + 200 mesh (0.149 to 0.074 mm) | U.S. Standard ASTM − 100 to + 200 mesh (0.149 to 0.074 mm) or other sizes which are <40 mesh (0.420 mm) |
| Leachant Volume | 10 cc/gram of sample mass | 10 cc/gram of sample mass or variable volume/sample mass |
| Temperature | 90 ± 2°C | 90 ± 2°C or other temperatures provided that any observed changes in reaction mechanism are noted |
| Atmosphere | Air | Air or $CO_2$ free air (optional) (see Section 10) |
| Type of System | Closed to transport | Open to transport in PFA TFE-fluorocarbon; Closed to transport in stainless steel |

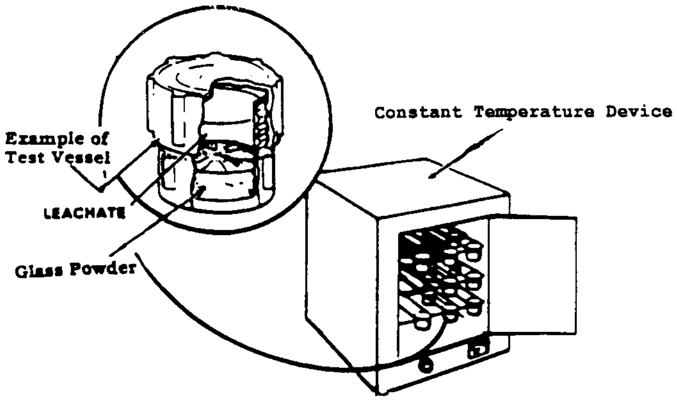

FIG. 1 Schematic of Test Apparatus

is easily reproducible, can be performed remotely if necessary, and can yield results rapidly. The glass does not need to be annealed prior to testing. In this test method the glass 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 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 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 cc/g of sample mass ( $m_{solid}$) 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 ± 2°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 the oven so that there is ample convection around the samples and even heat distribution (Fig. 1). After seven days± 3.4 h, or other optional test durations, the vessel is removed from the constant temperature device and cooled to ambient temperature. The pH is measured on an aliquot of the leachate and the temperature of the aliquot at the time of the pH measurement is also recorded. The remaining leachate is filtered and sent for analysis.

NOTE 1—Devitrified glasses 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 glasses as measured by elemental release. Accordingly, it may be applicable throughout manufacturing, research, and development.

5.1.1 Test Method A can specifically be used to obtain data to evaluate whether the chemical durability of waste glasses 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 glasses under various leaching conditions, for example, varying test durations, test temperatures, ratio of glass 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 $1 \times 10^5$ 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)**. In 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 waste glasses.

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 is 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 doses (>1 × 10 $^{5}$ rad of beta or gamma radiation) will not generally be experienced with hazardous, mixed or simulated nuclear waste glasses. PFA TFE-fluorocarbon vessels, available commercially, can be used in the absence of high radiation doses[9] since PFA TFE-fluorocarbon is chemically inert when properly cleaned **(9)**.

6.3 *Constant Temperature Devices*—Laboratory ovens or water baths capable of maintaining ±2.0°C uniformity throughout the entire interior of the device, including the samples, at the test temperature are to be used for sample leaching and sample drying. These devices must be equipped with an oven-temperature control.

6.4 *Conventional Oven*—Optional laboratory or drying ovens, capable of maintaining ±10°C, can be used for vessel cleaning and sample drying.

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

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.[10]

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 Glass*—A reference glass (see Ref **12**for example) of choice, similar in composition to the glass being tested, is to be tested in triplicate along with each batch of glasses tested (see Ref **13**). The reference glass 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 ($HNO_3$) and hydrofluoric acid (HF) for cleaning leach vessels.[11]

8.2 *ACS High Purity Acid*—Ultra high purity concentrated nitric acid ($HNO_3$) for acidification of leachates.[11]

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

8.4 *Solvents*—Ethanol 95 % pure and reagent grade acetone.

8.5 *ASTM Type I Water*—Type I water shall have a minimal electrical resistivity of 16.67 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

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[9] 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.

[10] Cellulose acetate filters such as Nalgene No. 190-2045, available from Savillex Corp., 6133 Baker Rd., Minnetonka, MN 55345, 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, or both, have been shown not to interfere with leachate analysis.

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

nuclear-grade resin,[12] then through a cellulose ester membrane having openings not exceeding 0.45 µm.[13]

8.5.2 Pass the purified water through an in-line conductivity cell to verify its purity. Alternatively, the water can be measured for all anions and cations to verify that there is less than a total dissolved solid content including soluble silica of 0.1 g/$m^3$ (see Specification 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.[11] 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 leach containers 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. For example, 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. The user is also cautioned about the attraction of steel for certain radionuclides such as americium, plutonium, and other redox sensitive species **(14)**.

10.1.1 Steel vessels represent "closed system" applications where the influx of $CO_2$ or $O_2$ into the leachate is not desired. The user is cautioned that the leachate concentrations and leachate pH in PFA TFE-fluorocarbon and steel vessels may be significantly different for some glasses due to differences in the leachate equilibration with $CO_2$ and $O_2$, that is, the differences in "open" and "closed" system conditions. **(15-23)**

10.1.2 It is recommended that 22 mL vessels[14] 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 waste glasses. 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 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 "open system" applications where the influx of $CO_2$ or $O_2$ into the leachant is either desirable or not of concern. The user is cautioned that the leachate concentrations and leachate pH in PFA TFE-fluorocarbon and steel vessels may be different due to differences in the leachate equilibration with $CO_2$ and $O_2$, 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 glasses with radiation doses above $1 \times 10^5$ rads beta or gamma.[9] The use of PFA TFE-fluorocarbon vessels for radiation doses $>10^5$ rads causes degradation of the PFA TFE-fluorocarbon, and subsequent uptake of $F^-$ and HF by the test solution. The presence of HF in the solution may change the rate of degradation of the glass due to the acidic conditions and $F^-$ ions that attack the glass **(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 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 % ethanol for approximately 5 min.

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[12] 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.

[13] 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 satisfactory.

[14] Vessels from Parr Instrument Co., 211 53rd St., Moline, IL 61265, have been found satisfactory.

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

12.1.4 Submerge the vessels and lids in 0.16 *M* $HNO_3$(1 weight% $HNO_3$) and heat to 90 ± 10°C for a minimum of 1 h. The gasket should not be in the lid during the $HNO_3$ cleaning step because of the possibility that small amounts of $HNO_3$ 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 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 % 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 $HNO_3$ and ASTM Type I water until the level of the radioactive element(s) of interest is(are) below the detectable level using the analytical method employed for concentration measurement of the leachate. Stainless steel vessels are also checked for silicon contamination before reuse. Used stainless steel containers for radioactive service (PCT Method A) shall be cleaned according to the following method:

14.1.1 Remove all glass by rinsing the vessel and lid with ASTM Type I water. Fill the vessel 80 to 90 % full with 0.16*M* $HNO_3$(1 weight % $HNO_3$). Reseal the vessel and place in 90 ± 10°C oven for a minimum of 16 h to dissolve (acid strip) any radionuclides adhering to the interior of the vessel.

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

14.1.3 If possible remove the gasket and discard. Gaskets that have been exposed to $HNO_3$ during cleaning or acid stripping of the vessel may be compromised because small amounts of $HNO_3$ may be trapped between the gasket and the lid. Rinse vessels and lids thoroughly with deionized water and then with ASTM Type I water at ambient temperature. Extreme caution should be exercised so that the inside of the vessel is not contaminated with radioactivity that may have contacted the outside of the vessel.

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

14.1.5 Remove vessels from oven, take one aliquot of water from each vessel and measure the pH (see Test Methods 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 $>0.1$ 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 $>0.1$ 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 ± 10°C for a minimum of 16 h and store in a clean environment until needed.

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

15.1 When stainless steel vessels are reused subsequent to their use with radioactive mixed waste specimens, residual contamination may be present. The vessels shall be cleaned before reuse by cleaning with $HNO_3$ and ASTM Type I water until the level of the radioactive element(s) of interest is(are) below the detectable level using the analytical method employed for concentration measurement of the leachate. Stainless steel vessels are also checked for silicon contamination before reuse. Used stainless steel containers for PCT Test Method B shall be cleaned according to the following method:

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

15.1.2 Remove the gasket from the lid and discard. Gaskets that have been exposed to $HNO_3$ during cleaning or acid stripping of the vessel may be compromised because small amounts of $HNO_3$ may be trapped between the gasket and the lid.

15.1.3 Soak the vessels and lids in 0.16 *M* $HNO_3$(1 weight % $HNO_3$) at 90 ± 10°C for 1 h.

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

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

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

15.1.7 Remove vessels from oven, take one aliquot of water from each vessel and measure the pH (see Test Methods 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 $>0.1$ $g/m^3$ 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 $>0.1$ $g/m^3$ silicon criteria cannot be achieved after three repetitions of 15.1.4 through 15.1.7, then repeat the cleaning and testing method starting at 15.1.2.

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

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

16.1 New PFA TFE-fluorocarbon vessels are cleaned before use with NaOH and ASTM Type I water to remove any free fluoride from the interior surfaces.[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± 10°C oven for at least seven days.

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

16.1.5 Remove the vessels from the oven after the seven days and allow 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 ± 10°C oven for a minimum of 16 h.

16.1.11 Remove vessels from oven. Allow vessels to cool to ambient temperature. Take an aliquot of water from each vessel and measure the pH (consult Test Methods 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 $>0.5$µ 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 ± 10°C for a minimum of 16 h, and store in a clean environment until needed.

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

17.1 When PFA TFE-fluorocarbon vessels are reused, residual contamination from the glasses tested may be present. The vessels shall be cleaned before reuse by cleaning with $HNO_3$ and ASTM Type I water. As a precaution fluoride contamination should continue to be checked for the first five uses of a given PFA TFE-fluorocarbon vessel. Used PFA TFE-fluorocarbon containers shall be cleaned according to the following method:

17.1.1 Remove all glass from the vessels by rinsing both the vessels and lid with ASTM Type I water.

17.1.2 Soak vessels and lids in 0.16 *M* $HNO_3$(1 weight % $HNO_3$) at 90 ± 10°C for approximately 1 h.

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

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

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

17.1.6 Remove vessels from oven, take an aliquot of water from each vessel and measure the pH (see Test Methods 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 $>0.5$µ g/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 must be handled with clean equipment and stored in clean containers. For highly radioactive glass, 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 may either be fabricated individually or taken from larger samples of glass (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.

19.3 *Choice of Sample Mass*—The reference ratio of leachant volume to sample mass ($V_{soln}/m_{solid}$) is 10± 0.1 cc/g. The volume of leachant is constrained by the volume of the leach vessel chosen and the need to minimize sample size when dealing with highly radioactive glasses. For example, 1.5 g of sample can be tested in 15 mL of leachant contained in a 22 mL steel vessel. Samples must be ≥1 g.

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*—If the glass is not fully oxidized (redox sensitive), the user is cautioned that grinding the glass in advance of the test may cause the glass to oxidize and may alter the leachate results. Redox sensitive glasses 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 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 glass needed to perform the PCT in triplicate in order to have enough glass to test.

19.5.1 Transfer glass fragments into a clean manual or mechanical grinder. Clean the grinder prior to crushing a different type of glass. 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 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 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, or both, 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. If a sieve is torn or deformed, discard the sieve and use a new sieve. Transfer crushed glass to the clean nest of sieves. No more than 100 g of crushed glass should be placed on an 8 in. diameter sieve (see Test Methods C 225 and C 371 (see Note 3)). Scaling the amount of glass to the exposed surface area of the sieve, indicates that no more than 50 g of crushed glass 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 glass should be placed on a 100 mesh (0.149 mm) sieve. A200 mesh (0.074 mm) sieve should be under the 100 mesh sieve with a catch pan below.

NOTE 3—The variability of the sample surface area to the solution volume has the largest effect on the precision of the test **(25)**. Particle size analysis of samples sieved with more than 100 g of glass on an 8 in. sieve has indicated a wider gaussian particle size distribution than for samples with <100 g of glass 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 device[15] 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).

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[15] The Ro-Tap Testing Sieve Shaker, supplied by W.S. Tyler Co., Mentor, OH 44060, is suitable.

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 glass 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 glass is light colored tap the sieve on dark paper. For dark glass, 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 glass 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 glass powder passes through the 200 mesh sieve.

19.5.7 Transfer the − 100 to + 200 mesh fraction of the sieved glass 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 fragments again or repeat 19.5.1. When new glass 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*:

19.6.1 Washing of the − 100 to + 200 mesh glass to remove adhering fines is required for homogeneous glasses (see Fig. 2). However, devitrified glasses may contain soluble secondary phases that might be substantially removed from the glass grains during the washing process. This possibility must be addressed when the test is used to determine the effect of devitrification on test leachate concentrations. Secondary phases present should be identified and either their potential dissolution rates relative to the bulk glass 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.

19.6.2 Place sieved glass in a clean glass beaker that will hold about 2.5 times the sample volume. For example if 15 to 20 g of sieved glass 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. 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 glass-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.

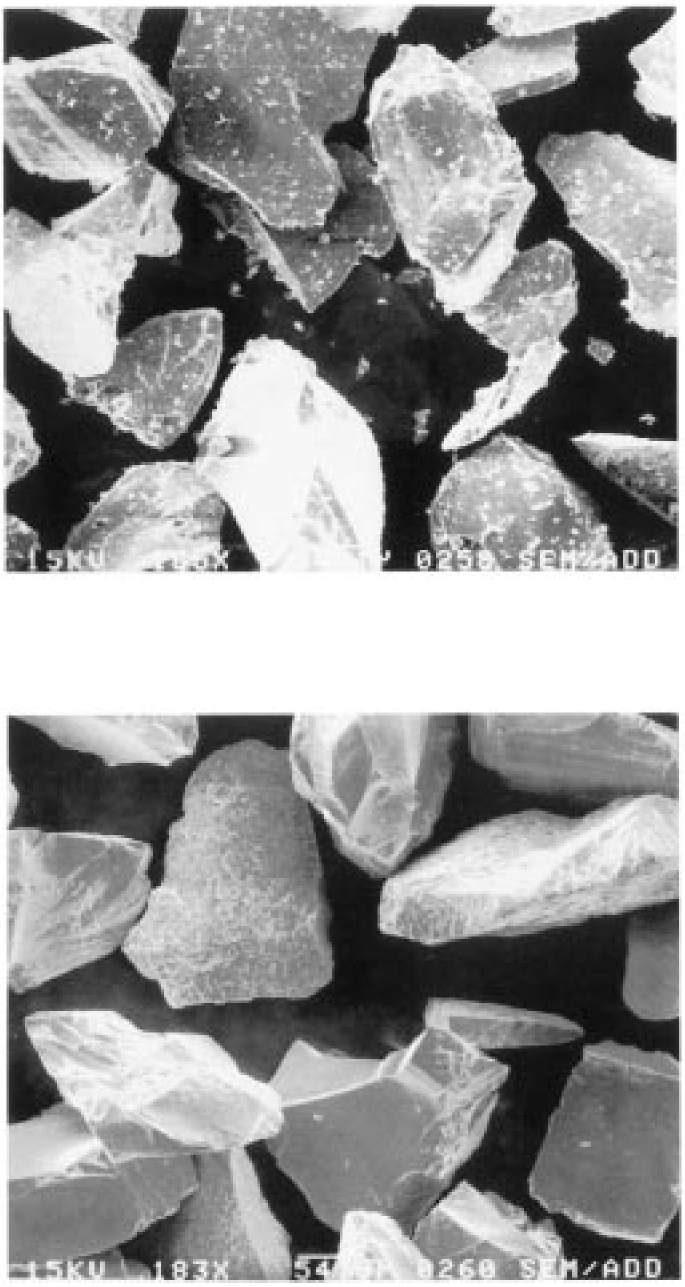

**FIG. 2 Adherent fines on − 100 to + 200 mesh glass particles before washing (Top). Glass particles after washing in ASTM Type I Water and Ethanol (Bottom)**

19.6.8 Repeat 19.6.6 and 19.6.7.

19.6.9 Forcibly add ethanol from a squirt bottle to the glass. 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 glass in a 90 ± 10°C oven overnight to dry. If possible, examine the dried glass under a microscope to ensure that the fines have been removed and that there are no metal filings from the grinding. Use a magnetic tweezer to move the glass 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 waste glass do not discard the sample and proceed to 19.6.13.

19.6.13 Store dried acceptable glass in a clean, sealed and labeled container in a dessicator until use. Use within three months. If the glasses have not been stored in a desiccator or they have been stored in a dessicator for over three months they must be redried at 90 ± 10°C overnight. Drying the glass 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 Glass for PCT Test Method A

20.1 The reference glass should be prepared at the same time as the "set" of unknown glasses being tested. The same person should prepare the reference glass using the same equipment that is used for the unknown glasses being tested.

20.1.1 *Reference Glass Handling*—same as 19.1.

20.1.2 *Reference Glass Sample Size*—same as 19.3.

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

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

## 21. Procedure—PCT Test Method A

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

21.2 *Number of Reference Glass Replicates*—A standard glass 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 glass. 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 16.67 MΩ·cm at 25°C. Alternatively, the water can be measured for all anions and cations to verify that there is less than a total dissolved solid content including soluble silica of 0.1 $g/m^3$(see Specification D 1193 and Terminology D 1129). Record the resistivity of the water collected on each log sheet including those for each sample glass, each reference glass, and each blank.

21.4.2 Calibrate the pH meter. Determine the pH of an aliquot of ASTM Type I water collected. 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 initial 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 weight on a sample log sheet like that given in Appendix X2.

21.4.5 Place the desired amount of prepared glass in the clean leach vessel. The glass may be preweighed prior to placing it in the vessel. If the glass is preweighed record the weight of the glass. Replace the lid and reweigh the vessel, lid and sample. Record this composite weight. If the glass 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 glass on a sample log sheet.

21.4.6 Add ASTM Type I water equivalent to ten times the mass of glass added as calculated in 21.4.5 so that ( $V_{soln}/m_{solid}$) = 10± 0.1 mL/g. Swirl to wet the glass. Cap and seal the leach container and reweigh. Record the total 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 glass vessels, and blanks should be placed immediately into a preheated 90 ± 2°C oven. The seven day ±3.4 h test period starts at this time. Record the date and time (d:h:min) on a sample log sheet and on the recording device which monitors the oven temperature.

21.4.8 The testing period shall be controlled to within± 2 % of the total time period of the test. 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 from the oven on a sample log sheet and on the recording device which periodically monitors oven temperature. 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 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, 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 must be analyzed for boron, silicon, and all alkali elements present in the glass at >1 weight %, for example, sodium, lithium, and potassium. Acidify the aliquots drawn for cation analysis with concentrated ultra high purity $HNO_3$ equal to 1 volume % of the aliquot volume (see Note 5). Perform cation analyses and include acidified multielement solution standards. For radioactive glasses, submit appropriately acidified aliquots for all desired radiochemical analyses. Note the analytic service identification number (if any) on the sample log sheet along with the analysis requested.

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

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

21.4.15 Measure cation and anion concentrations of glass leachates, standard glass 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.

21.4.16 Analysis of the solids on the filter or the remaining solid glass sample is optional. If solids analysis is not desired, the filter and solid glass 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 ≤90± 2°C. A temperature of 90 ± 2°C will only drive off adsorbed moisture and not water of hydration. After drying, store in a clean container or analyze (e.g., consult Test Methods C 1317 and C 1342).

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

### 22. Sample Preparation for PCT Test Method B

22.1 *Sample Handling*—All glass must be handled with clean equipment and stored in clean containers.

22.2 *Choice of Appropriate Sample*—Samples of glass may either be fabricated individually or taken from larger samples of glass (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*—The recommended ratio of leachant volume to sample mass ($V_{soln}/m_{solid}$) is 10± 0.1 mL/g. Other $V_{soln}/m_{solid}$ can be used. The volume of leachant is constrained by the volume of the leach vessel chosen. Samples must be ≥1 g.

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*—If the glass is not fully oxidized (redox sensitive), the user is cautioned that grinding the glass in advance of the test may cause the glass to oxidize and may alter the leachate results. Redox sensitive glasses 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 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 glass needed to perform the PCT in triplicate.

22.5.1 Transfer glass fragments into a clean manual or mechanical grinder. Clean the grinder prior to crushing a different type of glass. 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 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 should be cleaned by flushing them with deionized water from all directions. Dry immediately with high pressure air or in an oven. Do not use solvents or high temperatures, or both, 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. If a sieve is torn or deformed discard the sieve and use a new sieve. Transfer crushed glass to the a clean nest of sieves. No more than 100 g of crushed glass 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 to the exposed surface area of the sieve, indicates that no more than 50 g of crushed glass 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 glass 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 on an 8 in. sieve has indicated a wider Gaussian particle size distribution than for samples with <100 g of glass 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 device[15] 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 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 is light colored tap the sieve on dark paper. For dark glass, for example, 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 glass 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 glass powder passes through the smallest mesh sieve.

22.5.7 Transfer the fraction of the sieved glass 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 fragments of glass lying on top of the largest sieve screen or repeat 22.5.1. When new glass 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*:

22.6.1 Washing of the sized mesh glass to remove adhering fines is required for homogeneous glasses (see Fig. 2). However, devitrified glasses may contain soluble secondary phases that might be substantially removed from the glass grains during the washing process. This possibility must be addressed when the test is used to determine the effect of devitrification on test leachate concentrations. Secondary phases present should be identified and either their potential dissolution rates relative to the bulk glass 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.

22.6.2 Place sieved glass in a clean glass beaker that will hold about 2.5 times the sample volume. For example if 15 to 20 g of sieved glass 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 glass-water mixture 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 ethanol from a squirt bottle to the glass. 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.

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 glass under a microscope to ensure that the fines have been removed and that there are no metal filings from the grinding. Use a magnetic tweezer to move the glass 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 waste glass do not discard the sample and proceed to 22.6.13.

22.6.13 Store dried acceptable glass in a clean, sealed and labeled container in a desiccator until use. Use within three months. If the glasses 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 ± 10°C overnight. Drying the glass 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 Glass for PCT Test Method B

23.1 The reference glass should be prepared at the same time as the “set” of unknown glasses being tested. The same sample mesh size, $V_{soln}/m_{solid}$, type of vessel, test duration, and test temperature as the samples being tested should be used. The same person should prepare the reference glass using the same equipment that is used for the unknown glasses being tested.

23.1.1 *Reference Glass Handling*—Same as 22.1.

23.1.2 *Reference Glass Sample Size*—Same as 22.3.

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

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

## 24. Procedure—PCT Test Method B

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

24.2 *Number of Reference Glass Replicates*—A standard glass 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 glass. 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 16.67 MΩ·cm at 25°C. Alternatively, the water can be measured for all anions and cations to verify that there is less than a total dissolved solid content including soluble silica of 0.1 $g/m^3$(see Specification D 1193 and Terminology D 1129). If ASTM Type I water is used record the resistivity of the water collected on each log sheet including those for each sample glass, each reference glass, 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 an aliquot of the leachant. Pour the leachant into a sealed cleaned vessel for transport. Keep the leachant sealed until use. If ASTM Type I water is used note that slow absorption of gaseous species from the air can cause the initial 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 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 weight on a sample log sheet like that given in Appendix X2.

24.4.5 Place the desired amount of prepared glass in the clean leach vessel. The glass may be preweighed prior to placing it in the vessel. If the glass is preweighed record the weight of the glass. Replace the lid and reweigh the vessel, lid and sample. Record this composite weight. If the glass 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 glass on a sample log sheet.

24.4.6 Add leachant equivalent to the $V_{soln}/m_{solid}$ chosen. A $V_{soln}/m_{solid}$ of 10± 0.1 mL/g is recommended. Swirl to wet the glass. Cap and seal the leach container and reweigh. Record the total 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 glass vessels, and blanks should be placed immediately into the oven preheated to the desired temperature. The recommended temperature is 90 ± 2°C. The desired test period starts at this time. The recommended test duration is seven days ±3.4 h. Record the date and time (d:h:min) on a sample log sheet and on the recording device that monitors the oven temperature.

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

24.4.9 The testing period shall be controlled to within± 2 % of the total time period of the test. 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 from the oven on a sample log sheet and on the recording device which continuously monitors oven temperature. 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. Alternatively, a vessel with a septum can be used for periodic sampling of the leachate while at the test temperature. For each aliquot of leachate removed record the date and time (d:h:min) at which the sample is removed from the oven on a sample log sheet and on the recording device that periodically monitors oven 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, disregard the results of that test and use the remaining test results (minimum of two). If more than one replicate of the same glass 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 The solution must be analyzed for boron, silicon, and all alkali elements present in the glass at >1 weight %, for example, sodium, lithium, and potassium. Acidify the aliquots drawn for cation analysis with concentrated ultra high purity $HNO_3$ equal to 1 volume % of the aliquot volume (see Note 9).

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

24.4.13.1 Perform cation analyses and include acidified multielement solution standards. Note the analytic service identification number (if any) on the sample log sheet along with the analysis requested.

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

24.4.15 Measure cation and anion concentrations of glass leachates, standard glass 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.

24.4.16 Analysis of the solids on the filter or the remaining solid glass sample is optional. If solids analysis is not desired, the filter and solid glass 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 ≤90± 2°C. A temperature of 90 ± 2°C will only drive off adsorbed moisture and not water of hydration. After drying, store in a clean container or analyze (e.g., consult Test Methods C 1317 and C 1342).

## 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 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 was present in the test. Leachate concentrations, especially those for the major soluble elements in the glass (lithium, boron, sodium, potassium, and silicon) 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 contaminant 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 (lithium, boron, sodium, potassium, and silicon) are calculated for the test glasses and the standard glass. Standard glass leachate concentrations and pH values should be control charted. Concentrations from each new set of standard glass analyses should be compared to previous standard glass test responses. If the current standard glass data set 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 glass, then the data for all the samples must be bias corrected before it can be used. 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)**.

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.

25.2.3 Calculate the corrected leachate concentration for each sample by subtracting the average blank concentration from the measured concentration. Note that for a valid test, the blank concentrations are to be <10 % of the leachate concentrations.

25.2.4 Calculate the mean concentration for each valid element by dividing the sum of the corrected concentrations by the number of tests performed.

25.2.5 Calculate the standard deviation for the corrected leachate concentrations for valid element concentrations.

## 26. Report

26.1 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 (e.g., consult Test Methods C 1317 and C 1342).

26.2 All data should be recorded in a retrievable manner.

26.3 Deviations from the test method and the expected effect on the results should be discussed.

## 27. Precision and Bias

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.

27.1.2 *PCT Method A: Within-Laboratory Precision*—There is no round robin data involving three or more laboratories to support a statement concerning repeatability and reproducibility for PCT Test Method A in stainless steel vessels. However, data from two separate laboratories have been used to determine the within laboratory precision for remote radioactive operation (27.1.2.1 through 27.1.2.2). Within laboratory precision has also been determined for "hands on" operation with a non-radioactive glass (27.1.2.3).

27.1.2.1 *Radioactive Glass 200R* (a radioactive borosilicate glass containing U-235 neutron fission and activation products)—Within-laboratory standard deviations for boron 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 standard deviations were 0.7 and 1.1 %. Each laboratory performed triplicate tests on each glass.

27.1.2.2 *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.

27.1.2.3 *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*—There is no round robin data from three or more laboratories to support a statement concerning between laboratory precision for PCT Test Method A in stainless steel vessels using radioactive glass. Data from two separate laboratories have been used to determine the between laboratory precision for remote radioactive operation (27.1.3.1 through 27.1.3.2).

27.1.3.1 *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.

27.1.3.2 *Radioactive Glass 165/42* (a radioactive borosilicate waste glass containing U-235 neutron fission and activation products)—Between-laboratory relative standard deviation for boron and silicon was 14 % using washed − 100 to + 200 mesh glass in ASTM Type I water for seven days ±3.4 h (Data in Ref **27**, Table 4). Each laboratory performed triplicate tests on each glass.

27.1.4 *PCT Method B—Within-Laboratory Precision for PFA TFE-fluorocarbon Vessels*:

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)**. Each of the three laboratories analyzed the glass in triplicate.

27.1.5 *PCT Method B—Between-Laboratory Precision*:

27.1.5.1 *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 % and for silicon was 10.2 % 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 seven laboratories analyzed each glass in triplicate once a week for three weeks and each leachate was analyzed twice. Each of the seven laboratory populations are, therefore, eighteen (18) test results.

27.1.5.2 *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 19.8 % and for silicon was 18.3 % 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 seven 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.

27.2 *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 SURFACE AREA

X1.1 Glass particles are neither spherical, tabular, nor square (see Fig. 2). In order to calculate exposed glass surface area from the mass of glass used, one must assume a given geometric shape. If the glass 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 ($1.12 \times 10^{-4}$ m) introduces no significant error. For this example the glass density (e.g., 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

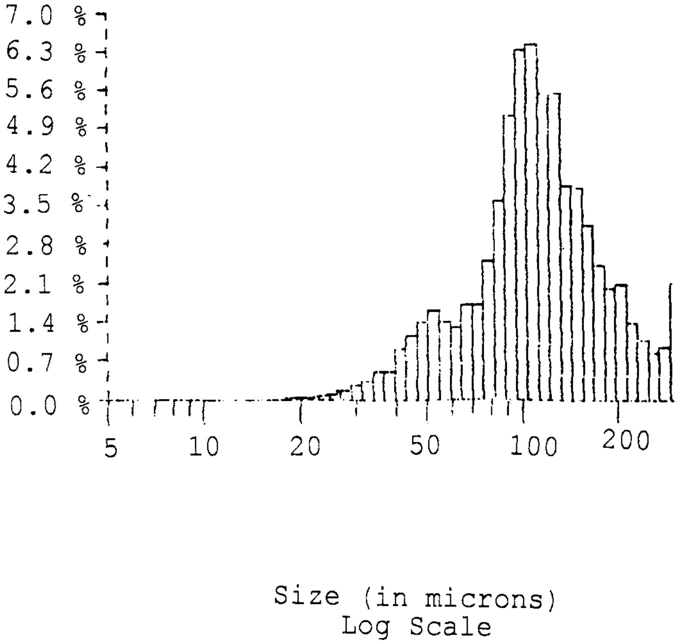

**FIG. 3 Example of the Particle Size Distribution on the –100 to +200 Mesh Sieve**

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

where $d$ is the average particle diameter, $1.12 \times 10^{-4}$ 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, $3.90 \times 10^{-8}$ m$^2$ and $7.25 \times 10^{-13}$ m$^3$, respectively. Since the assumed glass density is 2.7 g/cc or $2.7 \times 10^6$ g/m$^3$ and density is mass/unit volume, the mass of an average size particle can be determined as

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

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

This enables the calculation of the number of particles per gram of crushed glass, for example, there is 1 g/$1.96 \times 10^{-6}$ g = $5.11 \times 10^5$ particles in 1 g of − 100 to + 200 mesh glass. Each particle has a surface area of $3.90 \times 10^{-8}$ m$^2$ as given above. The total calculated surface area in 1 g of − 100 to + 200 mesh glass ($A$ per gram) is $A$ per gram = ($5.11 \times 10^5$ particles)($3.90 \times 10^{-8}$ m $^2$) = $1.99 \times 10^{-2}$ m$^2$. The calculated surface area in 4 g is $8 \times 10^{-2}$ m$^2$. As long as the glass density and glass 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 on ______________________________ by ______
2. Sample Washed on ______________________________ by ______
3. Sample Dried on ______________________________ by ______

*Vessel Preparation:*

1. Sample Vessel ID No. ______________________________ Batch Cleaning No. ______________________________
2. Vessel Cleaned on ______________________________ by ______
3. 1st Vessel Blank No. ______________________________ Batch Cleaning No. ______________________________
4. 2nd Vessel Blank No. ______________________________ Batch Cleaning No. ______________________________
5. Blanks Cleaned on ______________________________ by ______

*Run Data:*

| | Initial Conditions | Initials | Final Conditions | Initials | Change |
|---|---|---|---|---|---|
| 1. Type of solution | ____________ | ____________ | N/A | N/A | N/A |
| 2. Resistivity if ASTM Type I water | ____________ | ____________ | N/A | N/A | N/A |
| 3. pH/temperature (°C) of leachant | ____________ | ____________ | ____________ | ____________ | ____________ |
| 4. pH/temperature (°C) of buffer solutions | ____________ | ____________ | ____________ | ____________ | N/A |
| 5. pH meter ID No. | ____________ | ____________ | ____________ | ____________ | N/A |
| 6. Weight of empty vessel (g) | ____________ | ____________ | N/A | N/A | N/A |
| 7. Weight of vessel + lid + sample (g) | ____________ | ____________ | N/A | N/A | N/A |
| 8. Weight of sample (Item 7–6 g) | ____________ | ____________ | N/A | N/A | N/A |
| 9. mL of solution (Item 8 × 10) | ____________ | ____________ | N/A | N/A | N/A |
| 10. Weight of vessel + sample + lid + solution | ____________ | ____________ | ____________ | ____________ | (Note X2.1) |
| 11. test and temperature | PCT Version 7.0 at 90°C | | | ____________ | ____________ |
| 12. date, hour, min test started and ended | ____________ | ____________ | ____________ | ____________ | (Note X2.2) |
| 13. vessel lid retightened (Y/N) | ____________ | ____________ | N/A | N/A | N/A |

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

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

*Leachate Analyses:*

Acidifed with ________________________ on ______ by ______
Diluted with ________________________ on ______ by ______
Diluted factor ________________________ on ______ by ______

*Analytic Service ID's:*

Undiluted Cation Analysis No. ____________ for __________________
Diluted Cation Analysis No. ____________ for __________________
Corresponding Blank ID No. (Optional) ______ for __________________
Undiluted Anion Analysis No. ____________ for __________________
Diluted Anion Analysis No. ____________ for __________________
Solids Analysis No. ____________________ for __________________

*Remarks/Deviations/Comments:*

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