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NUCLEAR WASTE GLASS PRODUCT CONSISTENCY TEST (PCT) - VERSION 5.0 (U)

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ABSTRACT

Liquid high-level nuclear waste will be immobilized at the Savannah River Site (SRS) by vitrification in borosilicate glass. The glass will be produced in the Defense Waste Processing Facility (DWPF), poured into stainless steel canisters, and eventually disposed of in a geologic repository. In order to comply with the Waste Acceptance Preliminary Specifications (WAPS), the durability of the glass needs to be measured during production to assure its long term stability and radionuclide release properties.

A durability test, designated the Product Consistency Test (PCT), was developed for DWPF glass in order to meet the WAPS requirements. The response of the PCT procedure was based on extensive testing with glasses of widely different compositions. The PCT was determined to be very reproducible, to yield reliable results rapidly, and to be easily performed in shielded cell facilities with radioactive samples.

Version 5.0 of the PCT procedure is attached. This draft version will be submitted to ASTM subcommittee C26.13 on Repository Waste Package Materials Testing in January 1992.

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NUCLEAR WASTE GLASS PRODUCT CONSISTENCY TEST (PCT) - VERSION 5.0 (U)

INTRODUCTION

A durability test, designated the Product Consistency Test (PCT), has been developed for glasses produced in the Defense Waste Processing Facility (DWPF).[1] The test is designed to meet the requirements of the Waste Acceptance Preliminary Specifications (WAPS) 1.3 and 1.4.[2] Specification 1.3 requires the DWPF to demonstrate control of the radionuclide release properties of the final waste form. Changes in phase composition due to devitrification do not greatly alter the rate of release of material from the glass[3] of the type which will be produced in DWPF. However, the WAPS Specification 1.4 requires that the release properties of devitrified glass be similar to those determined in Specification 1.3. The DWPF is responsible for relating the results of the PCT to a repository site-specific release test, or, alternatively, for performing the repository site-specific release tests.

The PCT has been developed, in part, to satisfy the WAPS requirements by providing a test which is (1) sensitive to glass composition and homogeneity, and (2) has the potential to be related to repository site-specific release tests. The test was designed to provide confirmation of the consistency of DWPF glass while considering the following:

- sensitivity of the test to glass composition and homogeneity
- time necessary to demonstrate product quality
- ease of sample preparation for radioactive glass
- ease of test procedure for remote operation
- precision of the test results
- acceptance by waste form developers and repository projects

During PCT development, sample size was limited to 100-200 mesh (149-74μm) crushed glass because leaching of finer mesh sizes can cause overestimation of saturation concentrations, e.g. if finer powders are used, mass balance calculations need to be used to determine the maximum saturation concentration expected from a given particle size.[4] Fine particles also contribute larger errors to the estimation of the sample surface area than coarser sized samples. Moreover, use of a coarser mesh crushed glass simplifies sample preparation for radioactive service.

One test temperature, 90°C, was chosen for the PCT. This temperature is representative of the anticipated temperature in a repository because of the heat of decay of the radionuclides in DWPF waste glass. A single leachant, ASTM Type I water, was specified so that the test would be dominated by elemental species leached from the glass.

The V_{soln}/m_{solid} ratio for the PCT was chosen as 10 mL/g and test durations of 1, 3, 7, 14, and 28 days were evaluated. Seven days was chosen as the minimum test duration which optimized test precision but did not sacrifice discrimination.[1]

Leachate filtration to <0.45 μm was determined to improve the precision of the PCT. Filtering is advantageous because it removes colloidal species which would otherwise dissolve during the leachate acidification step and erroneously be measured as soluble elemental species. Filtering the leachate also removes the potential for fine glass particulates becoming entrained in the leachate acidification.[5] Such a dissolved particulate of glass would give an erroneously high soluble leachate concentration or contribute excessive radioactivity to the leachate.

PCT sample preparation specifies that the sieved glass should be washed in ASTM Type I water and absolute ethyl alcohol to remove electrostatically adhering fine particles. Comparisons of B.E.T. specific surface area measurements of alcohol washed and unwashed crushed basalt demonstrated that there was less than a 5% difference in the total surface area.[5] Other studies[6-9] have demonstrated that the <1μm fine particles only affect the initial non-linear kinetics of dissolution, e.g. the first 24 hour period. Thereafter, the fines are consumed with no further effect on the bulk dissolution. However, the amount of fines adhering to a glass sample is an uncontrollable quantity and, hence, sample washing was included in the PCT. Later experimental studies verified that sample washing improved the precision and the accuracy of the PCT.

An SRL internal round robin[1] and a seven laboratory external round robin were completed[10] in order to determine the precision and accuracy of the PCT. Confirmatory testing on radioactive samples was also performed.[11] These studies indicated that the PCT was very reproducible, yielded reliable results rapidly, and could be easily performed in shielded cell facilities with radioactive samples.

SUMMARY

Version 5.0 of the PCT procedure is attached. A draft of this version of the procedure was optimized after the external round robin.[10] This draft will be submitted to ASTM subcommittee C26.13 on Repository Waste Package Materials Testing in January 1992.

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1. C. M. Jantzen and N. E. Bibler, "**Product Consistency Test (PCT) and Test Protocol,**" USDOE Report DPST-87-575, Savannah River Laboratory, Aiken, SC (1987).

2. **Waste Acceptance Preliminary Specifications for the Defense Waste Processing Facility High-Level Waste Form**, U. S. Department of Energy, Office of Civilian Radioactive Waste Management, USDOE Document OGR/B-8, Washington, DC (1988).

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6. G.R. Holdren, Jr. and R.A. Burner, "**Mechanism of Feldspar Weathering I. Experimental Studies,**" Geochim. Cosmochim. Acta, 43, 1161-1171 (1979).

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9. V.N. Fleer "**The Dissolution Kinetics of Anorthite ($CaAl_2Si_2O_8$) and Synthetic Strontium Feldspar ($SrAl_2Si_2O_8$) in Aqueous Solutions at Temperatures Below 100°C: With Applications to the Geological Disposal of Radioactive Wastes**" Ph.D. Thesis, The Pennsylvania State University, University Park (1982).

10. G. F. Piepel, T. E. Jones, D. L. Eggett, G. B. Mellinger, "**Product Consistency Test Round Robin Conducted by the Materials Characterization Center - Summary Report**," USDOE Report PNL-6967, Materials Characterization Center, Battelle Pacific Northwest Laboratories, Richland, WA (1989).

11. N. E. Bibler and J. K. Bates, "**Product Consistency Leach Tests of Savannah River Site Radioactive Waste Glasses**," Scientific Basis for Nuclear Waste Management, XIII, V. M. Oversby and P. W. Brown (Eds.), Materials Research Society, Pittsburgh, PA, 327-338 (1990).

Attachment A

STANDARD TEST METHOD RELATIVE TO DURABILITY OF NUCLEAR WASTE GLASSES: THE PRODUCT CONSISTENCY TEST (PCT)

Version 5.0
October 30, 1991

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STANDARD TEST METHOD RELATIVE TO DURABILITY OF NUCLEAR WASTE GLASSES: THE PRODUCT CONSISTENCY TEST (PCT)

1. Scope

1.1 These Product Consistency Test methods 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 7 day crushed glass durability test at 90°C in a leachant of ASTM-Type I water. This method is static and conducted in stainless steel vessels. Test method A can specifically be used to evaluate whether the durability and elemental release characteristics of glasses have been consistently controlled during production. This 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, ratios of glass surface area to leachant volume, and leachant types. This method is static and can be conducted in stainless steel and/or PFA Teflon® vessels. Test Method B can specifically be used to evaluate the durability characteristics of homogeneous and/or devitrified glasses. This method is applicable to radioactive and simulated, waste glasses.

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

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 162 Standard Terminology of Glass and Glass Products[1]

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

C 1109 Standard Test Methods for Analysis of Aqueous Leachates from Nuclear Waste Materials using Inductively Coupled Plasma-Atomic Emission Spectrometry[2]

C 1174 Standard Practice for Prediction of the Long Term Behavior of Waste Package Materials Including Waste Forms Used in the Geologic Disposal of High-LevelNuclear Waste[3]

D 1129 Definitions of Terms Relating to Water[4]

D 1193 Specification for Reagent Water[5]

D 1293 Standard Test Methods for pH of Water[5]

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

D4327 Standard Test Method for Anions in Water by Ion Chromatography.[5]

E 691 Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

2.2 *Other Documents:*

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846A (latest version or equivalent)[6]

Product Consistency Test Round Robin Conducted by the Materials Characterization Center-Summary Report[7]

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

[3] *Annual Book of ASTM Standards, in press*

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

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

[6] *SW846A 3rd Edition, Revision 1, U.S. Environmental Protection Agency,Washington, DC, December, 1987.*

[7] *U.S. DOE Report PNL-6967* Battelle *Pacific Northwest Laboratory, Richland, WA, September 1989.*

Product Consistency Test (PCT) for DWPF Glass, Part I. Test Development and Protocol.[8]

3. Definitions

ASTM Type I water - purified water with a maximum total matter content of 0.1 g/m^3, a maximum electrical conductivity of 0.06μmho/cm at 25°C, a minimum electrical resistivity of 16.67 MΩ•cm at 25°C, and no detectable soluble silica (consult ASTM D 1193 and D 1129) [4]

chemical durability - the resistance of a glass to either physical or chemical change in an aqueous or humid environment. It is frequently evaluated after prolonged weathering or storing, in terms of chemical and physical changes in the glass surface, or in terms of changes in the contents of a vessel of aqueous solution (after ASTM C 162)[2]

closed system tests - a system that precludes the transport of matter either into or out of the system

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[11]

devitrified glass - glass that has crystallized during cooling and/or due to thermal heat treatment

leachant - the solution that is being used, or is intended for use in leaching

leachate - the solution resulting from a leach test

mixed waste glass - a glass comprised of glass forming additives and hazardous waste that can contain radioactive constituents

nuclear waste glass - a glass comprised of glass forming additives and radioactive waste

open system tests - a system that permits the transport of matter into or out of the system, e.g. O_2 and/or CO_2

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[4] *U.S. DOE Report DPST-87-575, Savannah River Laboratory, Aiken, SC 29808, July 30, 1987.*

diffusion into or out of the system.

radioactive - of or exhibiting radioactivity;[9] 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[10]

radioactivity - spontaneous nuclear disintegration with emission of corpuscular or electromagnetic radiation, or both (consult D 1129)[5]

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

set of samples - samples tested simultaneously in the same oven

simulated waste glass - a glass comprised of glass forming additives with non-radioactive and/or non-hazardous simulants of the chemical species in actual radioactive wastes, and/or in mixed nuclear wastes

4. Significance and Use

4.1 These test methods provides data useful for evaluating the chemical durability of glasses as measured by elemental release. Accordingly, it may be applicable throughout manufacturing, research, and development.

4.1.1 Test Method A can specifically be used to evaluate obtain data to evaluate whether the durability of waste glasses has have been consistently controlled during production (see Table I).

4.1.2 Test Method B can specifically be used to measure the durability of glasses under various leaching conditions, e.g. varying test durations, test temperatures, ratio of glass surface area to leachant volume (S/V ratio), and leachant types (see Table I). Data from this test may form part of the larger body of data that is necessary in the logical approach to long-term prediction of waste form behavior as described in ASTM C1174.

[9] *The American Heritage Dictionary, Houghton Mifflin, 2nd Edition, 1982.*

[10] *Websters New Twentieth Century Dictionary, Unabridged, 2nd Edition, The World Publishing Co., New York, 1973.*

Table I. Summary of Test Methods A and B

	Test Method A	Test Method B
Type of Glass	Radioactive Simulated	Radioactive Mixed Simulated
Usage	During production for rapid analysis	Scoping tests; Crystallization studies; Comparative waste form evaluation
Test Vessel	Unsensitized 304L stainless steel; vessels rated to > 0.5 Mpascals	Unsensitized 304L stainless steel or PFA Teflon®[11]; vessels rated to > 0.5 Mpascals
Test Duration	7 days	7 days or varying times
Leachant	ASTM Type I water	ASTM Type I water or other solutions
Condition	Static	Static
Sample Mass (g)	> 1	> 1
Particle Size	100-200 mesh	100-200 mesh or other mesh sizes which are < 40 mesh
Leachant Volume (cm3)	10 times sample mass	10 times sample mass or varying S/V ratios
Temperature (°C)	90	90 or other temperatures provided that the temperature does not change the leaching mechanism
Atmosphere (optional)	Air	Air or CO_2 removal
Type of System	Closed to transport	Open to transport in Teflon; Closed to transport in stainless steel

[11] *PFA Teflon® is perfluoralkoxy teflon; labware of PFA Teflon® is manufactured by Savillex® without plasticizers or organic additives*

5. Summary of the Test Methods A and B

Test Method A is the Product Consistency Test (PCT) which was developed specifically to test the durability of radioactive waste glasses during production. It can also be used to test simulated waste glasses. The 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 method the glass is crushed and sieved to 100-200 mesh, the particles are cleaned of adhering fines, and an amount of sized and cleaned glass that is greater than or equal to 1 gram is placed in a 304L stainless steel vessel. An amount of ASTM Type I water equal to 10 times the sample mass is added and the vessel is sealed. The vessel is placed to ensure ample convection around the samples and even heat distribution (Figure 1). After 7 days the vessel is removed from the oven and cooled to room 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 recorded as well. The remaining leachate is filtered and sent for analysis.

Test Method B is the Product Consistency Test (PCT) which was developed to test the durability of radioactive, mixed, or simulated waste glasses. The method 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 method the glass is crushed and sieved to 100-200 mesh or to the desired size range. Particles must be <40 mesh. The particles are cleaned of adhering fines, and an amount of sized and cleaned glass greater than or equal to 1 gram is placed in either a 304L stainless steel vessel or a PFA Teflon® vessel. An amount of ASTM Type I water equal to 10 times the sample mass is added and the vessel is sealed. Other sample mass-to-volume of solution (S/V) ratios are allowed and other leachants are allowed. The vessel is placed in a convection oven at 90°C. Other test temperatures are allowed as long as the leaching mechanism does not change over the temperature range being tested or evaluated. The samples must be placed to ensure ample convection around the samples and even heat distribution (Figure 1). After 7 days, or other optional test durations, the vessel is removed from the oven and cooled to room 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 recorded as well. The remaining leachate is filtered and sent for analysis.

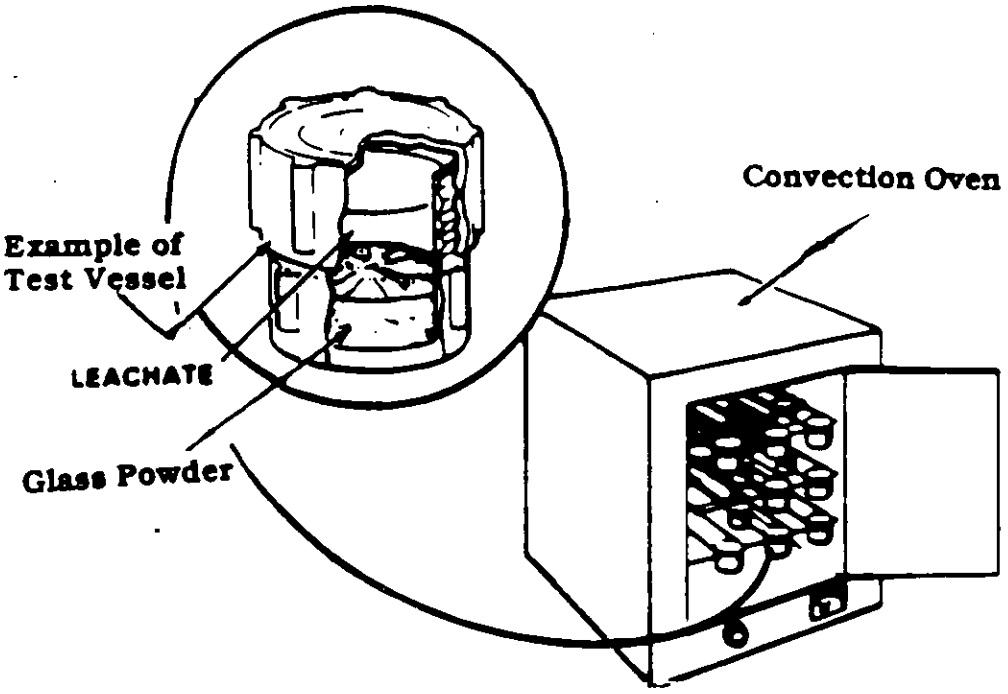

Figure 1. Schematic of Test Methods A and B

6. Apparatus

6.1 *Test Vessels for Method A* - The production test method requires the use of unsensitized 304L stainless steel leach vessels of > 20mL capacity designed to take internal pressures of >0.5 Mpascals without leaking (See Section 10).[12]

The stainless steel vessels require a gasket material in order to remain sealed. Teflon® gaskets, available commercially, are suggested since Teflon® is chemically inert and exposure to radiation fields up to 1 x 10^5 rad of beta or gamma radiation have been shown[13]

[12] *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, e.g. filling a 60mL 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.*

[13] *D.M. Strachan, "Effect of Gamma Irradiation on Simulated Waste Glass Leaching and on the Leach Vessel," J. Am. Ceram. Soc. 66[9], C-158-C-160 (1983).*

not to damage Teflon®. If higher radiation fields are present, it may be necessary to use special gaskets fabricated from metals such as copper, gold, lead or indium. High radiation fields will not be experienced with simulated waste glasses.

6.2 *Test Vessels for Method B* - Test Method B allows for the use of either unsensitized 304L stainless steel or PFA Teflon® leach vessels of > 20 mL capacity designed to take internal pressures of > 0.5 Mpascals without leaking (See Section 10).[12]

The stainless steel vessels require a gasket material in order to remain sealed. If radioactive glass is tested in stainless steel vessels with Teflon® gaskets the same constraints that are noted in Section 6.1 for radioactive usage in Test Method A apply.

High radiation fields will not be experienced with hazardous, mixed or simulated nuclear waste glasses. Teflon® vessels, available commercially, can be used in the absence of high radiation fields[13] since Teflon® is chemically inert when properly cleaned.[14]

6.3 *Convection Oven* - Laboratory ovens capable of maintaining ±2.0°C throughout the entire interior of the oven at the test temperature are to be used for sample leaching and sample drying. These ovens must be equipped with an over-temperature control.

6.4 *Conventional Oven* - Optional laboratory ovens, capable of maintaining ±10°C, can be used for vessel preparation.

6.5 *Temperature Measurement Device* - Resistance thermometers and/or thermocouples 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 hours.

6.6 *Balance(s)* - Any balance which will provide the following sensitivity: 0.25% of the largest and 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 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).

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[14] *H.M. Kingston and L. B. Jassie (Eds.), Introduction to Microwave Sample Preparation, Theory and Practice, ACS, Washington, DC, 263p , 1988.; J. Goodman and L. Mudrak, "Chemical Carryover from PFA Process Wafer Carriers, Solid State Technology, October, 1988.*

6.8 *Crushing Device* - Any mechanical or manual crushing device which will avoid iron (mild steel) contamination in the crushed glass specimen.[15] Grinding devices made of tungsten carbide, agate, sapphire, 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 pan, 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 which 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 0.45µm syringe filters of cellulose acetate.[16]

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, e.g. Inductively Coupled Plasma-Atomic Emission Spectrometry (consult ASTM C 1109 and/or EPA SW846A), Atomic Adsorption Spectrometry, Ion Chromatography (consult ASTM D 4327 and/or EPA SW846A), and/or Ion Selective Electrodes.

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[15] *G.L. McVay and C.Q. Buckwalter, "Effect of Iron on Waste Glass Leaching," J. Am. Ceram. Soc., 66, 170-174 (1983).*

[16] *Cellulose acetate filters such as Nalgene #190-2045 have been shown not to interfere with leachate analysis*

7. Calibrations

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

7.2 *Calibration and Standardization Schedule* -

7.2.1 *Temperature Measurement Devices* - Calibrate annually with standards traceable to NIST or an ice/boiling water bath.

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

7.2.3 *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. 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. Inasmuch as commercially available pH assemblies exhibit different degrees of measurement stability, conduct these checks at intervals of 30 minutes, unless it is ascertained that less frequent checking is satisfactory to ensure the performance described in ASTM D 1293.

7.2.4 *Water Purification System* - Calibrate 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 (consult ASTM D 1125).

8. Standards

8.1 *Reference Glass* - A reference glass of choice, similar in composition to the glass being tested, is to be tested in triplicate along with each batch of glasses tested. The reference glass composition should be traceable to NIST, or to a comparable source.

8.2 *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.

8.3 *pH Buffers* - commercial pH buffers or pH buffers made to the specifications given in ASTM D1293 which 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 stoppered sealed and replace at the expiration of shelf life, or sooner if a visible change is observed (consult ASTM D 1293).

8.4 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.

9. Reagents and Standards

9.1 *ACS Reagent Grade Acids* - Reagent grade nitric acid (HNO_3) and hydrofluoric acid(HF) for cleaning leach vessels.

9.2 *ACS High Purity Acid* - Ultra high purity concentrated nitric acid(HNO_3) for acidification of leachates.

9.3 *Reageant Grade NaOH* - Reageant grade NaOH for cleaning of new PFA Teflon® vessels.

9.4 *Solvents* - Ethyl Alcohol - 95% pure, reagent grade acetone.

9.5 *ASTM Type I Water* -Type I water shall have a minimal electrical resistivity of 16.67 MΩ•cm at 25° C (consult ASTM D1193).

9.5.1 The source water shall be purified, then passed through a deionizer cartridge packed with a mixed bed of nuclear-grade resin,[17] then through a cellulose ester membrane having openings not

[17] *A nuclear-grade resin mixture of the strong acid cation exchanger in the hydrogen form and the strong base anion exchanger in the hydroxide form with a one-to-one cation to anion equivalence ratio, 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.*

exceeding 0.45μm.[18]

9.5.2 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 of 0.1 g/m^3 and no detectable soluble silica (consult ASTM D 1193 and D 1129).

9.6 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. All leachants should be chemically analyzed to verify their composition before durability testing begins. All leachants should have a specified shelf life.

10. Choice of Test Vessel

10.1 *Stainless Steel Vessels* - Unsensitized 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 which could permeate the grain boundaries of the steel during fabrication and milling. The user is also cautioned about the attraction of steel for certain radionuclides such as Am, Pu, and other redox sensitive species.[19]

Steel vessels represent "closed system" applications where the influx of CO_2 or O_2 into the leachate is not desired.

It is recommended that 22mL Parr type vessels be used for the radioactive production application in Test Method A to minimize the amount of radioactive sample being handled.

10.2 *PFA Teflon® Vessels* - PFA Teflon® vessels may be used in Test Method B. PFA Teflon® vessels can be used for Test Method B for short-term durability testing with mixed or simulated nuclear waste glasses. The use of Teflon® vessels is recommended for test durations of < 28 days. Longer test durations are acceptable only if the

[18] *An in-line filter such as those made by the Millipore Corp., Bedford, MA 01730; Gelman Instrument Co., 600 S. Wagner Road, Ann Arbor, MI 48106; and Schleicher and Schuell, Inc., 540 Washington St., Keene, NH 10003, has been found satisfactory.*

[19] *W.J. Gray, "Effects of Radiation on the Oxidation Potential of Salt Brine, Scientific Basis for Nuclear Waste Management, XI, Materials Research Society Symposium Volume 112, p. 405 (1987).*

experimenter proves that the vessel is inert. The user should ensure that new Teflon® vessels are free from fluoride which is present as a free surface fluoride from vessel fabrication (see Section 16).

PFA Teflon® vessels represent "open system" applications where the influx of CO_2 or O_2 into the leachant is either desirable or not of concern.

PFA Teflon® vessels cannot be used in Test Method A and it is recommended that PFA Teflon® vessels not be used in Test Method B for glasses with radiation fields above 1 x 10^5 rads beta or gamma.[14] The use of PFA Teflon® vessels for radiation fields above>10^5 rads causes degradation of the Teflon®, and subsequent uptake of F- and HF by the test solution. The presence of HF in the solution causes accelerated degradation of the glass due to the acidic conditions and F- ions that attack the glass. [20]

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.

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.

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 I. This data will be maintained in a laboratory notebook for control purposes.

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[20] *N.E. Bibler, "Leaching Fully Radioactive SRP Nuclear Waste Glass in Tuff Groundwater in Stainless Steel Vessels," Nuclear Waste Management, II, D.E. Clark et. al. (Eds), Advances in Ceramics , 20, American Ceramic Society, Westerville , OH, 619-626 (1986).*

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

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

12.1 Degrease the vessels and lids (without the Teflon® gaskets) in acetone.

12.2 Clean the vessels and lids ultrasonically in 95% ethanol for ~ 5 minutes.

12.3 Rinse the vessels and lids 3 times with ASTM Type I water.

12.4 Submerge the vessels and lids in 0.16M HNO_3 (1 wt% HNO_3) and heat on a hotplate to 90°C for 1 hour.

12.5 Rinse the vessels 3 times with ambient temperature ASTM Type I water.

12.6 Submerge the vessels and lids in fresh ASTM Type I water for 1 hour at 90°C.

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

12.8 Fill the vessel 80-90% full of ASTM Type I water. Close the lid and leave in a 90°C oven for a minimum of 16 hours.

12.9 Remove the vessels from the oven, cool to room temperature, take a cooled aliquot of the water and measure the pH (consult ASTM D1293).

12.10 If the pH is not in the range 5.0-7.0, repeat steps 12.6 through 12.9.

12.11 If the 5.0-7.0 pH range cannot be achieved by 3 repetitions of steps 12.6 through 12.9 , then repeat the cleaning and testing method starting at Step 12.4.

12.12 Dry vessels and lids at 90±10°C for a minimum of 16 hours. Cool. If the vessels are not used immediately close the vessels and store in a clean environment until needed.

13. Cleaning of New Teflon Gaskets for Stainless Steel Vessels for PCT Test Methods A and B

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

13.1 Handle the gaskets only with clean tongs.

13.2 Clean each gasket ultrasonically in 95% ethanol for~10 minutes.

13.3 Clean each gasket under flowing ASTM Type I water at ambient temperature for ~3 minutes.

13.4 Bake each gasket in an oven at 200±10°C for a minimum of 4 hours.

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

13.6 Dry gaskets at 90±2°C for a minimum of 16 hours, and store in a clean environment until needed.

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

Used stainless steel containers for radioactive service (PCT Method A) shall be cleaned according to the following method:

14.1 Remove all glass by rinsing the vessel and lid with ASTM Type I water. Fill the vessel 80-90% full with 0.16M HNO_3. Reseal the vessel and place in 90±2°C oven for a minimum of 16 hours to acid strip any radionuclides adhering to the interior of the stainless steel vessel.

14.2 Check the acid stripped solution for radioactivity. Repeat step 14.1 until the radioactivity of the acid strip solution is minimal compared to that found when the glass was present.

14.3 Remove the gasket and discard. Rinse vessels and lids thoroughly with deionized water and then with ASTM Type I water at ambient temperature. 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.

14.4 Fill the vessel 80-90% full of fresh ASTM Type I. Close the lid and leave in a 90±2°C oven for a minimum of 24 hours.

14.5 Remove vessels from oven, take an aliquot of the water and measure the pH (consult ASTM D1293). Take another aliquot of the water and measure the radioactivity and the Si content of the solution.

14.6 If the pH is not in the range 5.0-7.0, **or** the measured radioactivity is not minimal, or Si is detected in the solution, repeat steps 14.3 through 14.5.

14.7 If the 5.0-7.0 pH range, the minimal radioactivity criteria, and/or the minimal Si criteria cannot be achieved by 3 repetitions of steps 14.3 through 14.6, then repeat the cleaning and testing method starting at Step 14.1.

14.8 Dry vessels, lids, and gaskets at 90±2°C for a minimum of 16 hours and store in a clean environment until needed.

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

Used stainless steel containers for non- radioactive service (PCT Test Method B) shall be cleaned according to the following method:

15.1 Remove all glass by rinsing the vessel and lid with ASTM Type I water at ambient temperature.

15.2 Soak vessels in 0.16M HNO_3 at 90°C for 1 hour. Do not submerge the stainless steel lids and their gaskets in the HNO_3 because of the possibility that small amounts of HNO_3 may be trapped between the gasket and the lid.

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

15.4 Put vessels and lids on a hotplate in ASTM Type I water at 90±2°C. Remove after 1 hour.

15.5 Fill the vessel 80-90% full of fresh ASTM Type I water. Close the lid and leave in a 90±2°C oven for a minimum of 24 hours.

15.6 Remove vessels from oven, take an aliquot of the water, measure the pH and the Si content (consult ASTM D1293).

15.7 If the pH of the aliquot is not in the range 5.0-7.0 or any Si is detected repeat steps 15.3 through15.6.

15.8 If the 5.0-7.0 pH range cannot be achieved or Si is dectected in the aliquot after 3 repetitions of steps15.3 through 15.6, then repeat the cleaning and testing method starting at Step 15.7.

15.9 Dry vessels, lids, and gaskets at 90±2°C for a minimum of 16 hours and store in a clean environment until needed.

16. Cleaning of New Teflon Vessels for PCT Test Method B

New Teflon® leach containers shall be cleaned according to the following method:

16.1 Rinse PFA Teflon® vessels and lids with fresh ASTM Type I water at ambient temperature.

16.2 Fill vessels 90% full with 5% NaOH solution.

16.3 Tighten lids and place vessels in a preheated 110±10°C oven for 7 days.

16.4 After 12-24 hours remove the vessels from the oven long enought to retighten the lids.

16.5 Remove the vessels from the oven after the 7 days and allow to cool to room temperature.

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

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

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

16.9 Repeat 16.7 and 16.8.

16.10 Dry vessels and lids at 90±2°C for a minimum of 16 hours, and store in a clean environment until needed.

16.11 Fill the Teflon® vessels about 90% full with fresh ASTM Type I water at ambient temperature. Close the vessels and leave in a

90±2°C oven for a minimum of 16 hours.

16.12 Remove vessels from oven. Allow Vessels to cool to room temperature. Take an aliquot of the water and measure the pH (consult ASTM D1293).

16.13 If the pH is in the 5.0 to 7.0 pH range, check the F^- concentration by measuring the F^- content of a second aliquot of the water.

16.14 If the pH is not in the range 5.0-7.0 or the F^- content is > 0.5µg/mL, repeat steps 16.1 to 16.13.

16.15 If the pH is above 7.0 repeat steps 16.7 to 16.13.

16.16 Dry vessels and lids at 90±2°C for a minimum of 16 hours, and store in a clean environment until needed.

17. Cleaning of Used Teflon® Vessels for PCT Test Method B

Used PFA Teflon® containers shall be cleaned according to the following method:

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

17.2 Soak vessels and lids in 0.16M HNO_3 (1wt% HNO_3) at 90±10°C for ~1 hour on a hotplate.

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

17.4 Put vessels and lids on a hotplate in fresh ASTM Type I water at 90±10°C. Remove after ~1 hour.

17.5 Fill the vessel 80-90% full of fresh ASTM Type I water at ambient temperature. Close the lid and leave in a 90±2°C oven for a minimum of 16 hours.

17.6 Remove vessels from oven, take an aliquot of the water and measure the pH (consult ASTM D1293) .

17.7 If the pH is in the 5.0 to 7.0 pH range, check the F^- concentration by measuring the F^- content of a second aliquot of the water.

17.8 If the pH is not in the range 5.0-7.0 or the F^- content is > 0.5µg/mL, repeat steps 17.4 to 17.7.

17.9 If the 5.0-7.0 pH range or the F^- content cannot be achieved by 3 repetitions of steps 17.4 to 17.7, then repeat the cleaning and testing method starting at Step 17.2.

17.10 Dry vessels and lids at 90±2°C for a minimum of 16 hours, and store in a clean environment until needed.

PCT Test Method A

18. Sample Preparation for PCT Test Method A

18.1 *Sample Handling* - All glass must be handled with clean equipment and stored in clean containers. For highly radioactive glass operations must be performed in a hot cell with manipulators: as much care as possible must be taken during these sample preparation steps.

18.2 *Choice of Appropriate Sample* - Samples of glass may either be fabricated individually or taken from larger samples of glass. The glass does not have to be annealed. Visually choose representative monolithic samples. Flush the sample surface with ASTM Type I water to remove potential surface contamination and dry before crushing.

18.3 *Choice of Sample Mass* - The reference ratio of leachant volume to sample mass (V_{soln}/m_{solid}) is 10 ± 0.1 mL/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 22mL steel vessel. Samples must be > 1 gram.

18.4 *Number of Sample Replicates* - A minimum of three replicate samples shall be used to provide estimates of experimental variability.

18.5 *Crushing and Sieving Glass* - If the sample has dimensions larger than 3/4", wrap the sample in a clean plastic bag and break it into smaller fragments with a hammer. For radioactive glass it may be necessary to use a steel mortar and pestle but caution must be exercised not to transfer mild steel particulates to the glass due to the known interactions of mild steel and glass in solution.[16] Crushing devices of 304L and 316L should be used to minimize these effects.

18.5.1 Transfer glass fragments into a clean manual or mechanical grinder of choice. Clean the grinder whenever a different glass sample is being crushed. Do not use mechanical grinders with steel blades unless they are known to be 304L or 316L stainless steel because of the known interactions of lower carbon containing steels and glass in solution. [16] If a small laboratory grinding mill such as a Tekmar® 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 ~1 to 1.5 cm before using the grinding mill. The sample basket of laboratory grinding mills can also be made of steel. If the sample basket becomes dull due to erosion of the steel, replace the sample basket.

18.5.2 Clean brass or stainless steel sieves, catch pan and lid before and after every use.[21]

18.5.3 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 placing the fragments on the 100 mesh (149 micron) sieve. The 200 mesh (74 micron) sieve should be under the 100 mesh sieve with a catch pan below.

18.5.4 Place the lid on the nest of sieves and sieve mechanically or manually for approximately 5 minutes.

18.5.5 Remove the 100 mesh sieve containing +100 mesh glass fraction. Then remove the 200 mesh sieve containing the -100 to +200 mesh fraction.

18.5.6 Tap the 200 mesh sieve lightly over contrasting colored paper. For example if the glass is light colored tap the sieve on dark

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[21] *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 and/or high temperatures as the brass mesh has a protective film to inhibit corrosion.*

paper. For dark glass, e.g. black nuclear waste glass, tap the sieve on white paper. If a significant amount of powder appears on paper, repeat steps 18.5.4 through 18.5.6 until a minimal amount of glass powder passes through the 200 mesh sieve.

18.5.7 Transfer the -100 to +200 fraction of the sieved glass into a clean container labeled with the sample identification. The date and the name of the person preparing the sample should also appear on the container.

18.5.8 If additional material is needed, recrush the +100 mesh size fragments or repeat step 18.5.1. When new glass fragments have been prepared repeat steps 18.5.3 through 18.5.4.

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

18.6 *Washing the 100-200 Mesh Glass* -

18.6.1 Place sieved glass in a clean glass beaker which will hold about 2.5 times the sample volume. For example if 15-20 grams of sieved glass is used, a 50 mL glass beaker should be used.

18.6.2 Forcibly add ambient temperature ASTM Type I water from a squirt bottle to the glass. The volume of water added should be about twice the sample volume. For example, if 15-20 grams of glass is used then add 25-30 mL of ASTM Type I water. During water addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

18.6.3 Allow the glass-water mixture to settle ~15 seconds, then decant off the water.

18.6.4 Repeat steps 18.6.2 to 18.6.3.

18.6.5 Repeat step 18.6.2.

18.6.6 Prepare the ultrasonic cleaner by filling with water to ~1 cm. Place the beaker from step 18.6.5 in the ultrasonic cleaner for 2 minutes. After removing the beaker from the cleaner, decant the water from the beaker and discard.

18.6.7 Repeat 18.6.6.

18.6.8 Forcibly add absolute alcohol from a squirt bottle to the glass. The volume of alcohol added should be about twice the sample volume. For example, if 15-20 grams of glass is used add 25-30 mL of absolute alcohol. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

18.6.9 Place the beaker from step 18.6.8 in the ultrasonic cleaner for 2 minutes. After the 2 minutes decant the alcohol from the beaker.

18.6.10 Repeat step 18.6.9 two more times.

18.6.11 Put the beaker full of cleaned glass in a 90±2°C ovenovernight to dry. Store glass in a clean, sealed and labeled container in a dessicator until use. Use within 3 months. If the glasses have not been stored in a dessicator or they have been stored in a dessicator for over 3 months they must be redried at 90±2°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.

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

19. *Preparation of Reference Glass for PCT Test Method A*

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.

19.1 *Reference Glass Handling* - same as18.1.

19.2 *Reference Glass Sample Size* - same as 18.3.

19.3 *Number of Standard Reference Glass Replicates* - same as 18.4.

19.4 *Crushing, Sieving, and Washing of Reference Glass* - same as Sections 18.5 and 18.6 .

20. *Procedure - PCT Test Method A*

20.1 *Number of Sample Replicates* - All tests for each glass should be carried out at least in triplicate (see Section18.4).

20.2 *Number of Reference Glass Replicates* - A standard glass test shall be run at least in triplicate as part of each "set" of samples.

20.2.1 A "set" of samples is considered to be those which are tested simultaneously in the same oven.

20.3 *Number of Vessel Blanks* - A blank is considered to be a cleaned test vessel which 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.

20.3.1 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 I.

20.4 *Leaching Method* - Each sample, standard, and blank shall be tested according to the following method:

20.4.1 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. Record the resistivity of the water collected on each log sheet including those for each sample glass, each reference glass, and each blank.

20.4.2 Calibrate the pH meter. Determine the pH of an aliquot of ASTM Type I water collected. Put the water in a sealed cleaned vessel for transport to the shielded cell. Keep to water 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 ASTM D1125). 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 I. Initial all measurement entries on the sheet. Discard the aliquot of ASTM Type I water used for the pH measurement.

20.4.3 Standardize the balance according to Section 7.2.2. It is recommended that a user's log of the balance standardization be kept. Record the balance identification number and the annual calibration date.

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

20.4.5 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.

20.4.6 Add ASTM Type I water equivalent to 10 times the mass of glass added as calculated in 20.4.5 so that $(V_{soln}/m_{solid})= 10\pm0.1$ 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 the same amount of water but no sample.

20.4.7 The "set" of samples including the reference glass vessels, and blanks should be placed immediately into a preheated 90±2°C oven. The 7 day test period starts at this time. Record that date and time (d:h:min) on a sample log sheet and on the recording device which continuously monitors the oven temperature.

20.4.8 The testing period shall be controlled to within plus or minus 1% of the total time period of the test. At the conclusion of the test remove the leach container from the oven and allow the container to cool to room temperature. 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 20.4.9 to 20.4.12 must be done as soon as the leachate has cooled to ambient temperature.

20.4.9 Check the balance calibration according to Section7.22. 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, abandon 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, abandon the triplicate tests and the test specimens. Repeat the test in triplicate starting with new test specimens.

20.4.10 For remote operation with radioactive glass, the leachate will need to be removed from the radioactive cell to minimize contaminating the solution. Carefully decant the leachate into a clean transport vessel and transfer this vessel to a radiochemical hood. Once in the radiochemical hood carefully transfer the solution from the transport vessel to another clean vessel to avoid contamination.

20.4.11 Calibrate the pH meter (see Section 7.2.3 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.

20.4.12 Draw a sufficient amount of the remaining leachant through a 0.45 μm syringe filter into a clean disposable syringe.[22,9] Note that more than one syringe filter may be necessary per sample. Remove the filter and transfer the contents of the syringe into a clean specimen bottle[23] for cation analysis. For poorly durable glasses the solution will need to be diluted before acidification (Section 20.4.13) in order to prevent gellation of the leachate. 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 (Section 20.4.14).

20.4.13 The solution must always be analyzed for Na, B, Si, Li, and K. Acidify the aliquots drawn for cation analysis with concentrated

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[22] *E. Vernaz, T. Advocat, and J.L. Dussossoy, "Effects of the SA/V Ratio on the Long-Term Corrosion Kinetics of R7-T7 Glass," Nuclear Wte Management, III, G.B. Mellinger (Ed.), Ceramic Transactions, v.9, Am. Ceramic Society, Westerville, OH, P. 175-185 (1990).*

[23] *Sample vials can be cleaned by boiling specimen bottles and caps for 1 hour 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.*

ultra high purity HNO_3 equal to 1% of the aliquot volume. [24] 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 on the sample log sheet along with the analysis requested.

20.4.14 Anion analysis is optional but is strongly recommended. Send the aliquot drawn for anion analysis but do not acidify the leachants. Perform anion analysis and include unacidified multielement solution standards. Note the analytic service identification number on a sample log along with the analysis requested.

20.4.15 Measure cation and anion concentrations of glass leachates, standard glass leachates, blanks, and multielement solution standards (consult ASTM C 1109 and D 4327). The short-term precision of these analytic methods at concentrations at least 100 times the detection limit range from 0.3 to 2% relative standard deviation.[25] Precision degrades with decreasing concentration to approximately 25% relative standard deviation at approximately two times the detection limit. [25]The detection limits for each analysis should accompany the reported results.

20.4.16 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, e.g., visible changes in color, agglomeration, and gelatinization. Wash the specimen powder from the leach container with pure water onto a clean watch glass and dry at not > 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.

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[24] *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*

[25] *ASTM C1109*

PCT Test Method B

21. Sample Preparation for PCT Test Method B

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

21.2 *Choice of Appropriate Sample* - Samples of glass may either be fabricated individually or taken from larger samples of glass. The glass does not have to be annealed. Visually choose representative monolithic samples. Flush the sample surface with ASTM Type I water to remove potential surface contamination and dry before crushing.

21.3 *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 gram.

21.4 *Number of Sample Replicates* - A minimum of three replicate samples shall be used to provide estimates of experimental variability.

21.5 *Crushing and Sieving Glass* - If the sample has dimensions larger than 3/4", wrap the sample in a clean plastic bag and break it into smaller fragments with a hammer. For radioactive glass it may be necessary to use a steel mortar and pestle but caution must be exercised not to transfer mild steel particulates to the glass due to the known interactions of mild steel and glass in solution.[16] Crushing devices of 304L and 316L should be used to minimize these effects.

21.5.1 Transfer glass fragments into a clean manual or mechanical grinder of choice. Clean the grinder whenever a different glass sample is being crushed. Do not use mechanical grinders with steel blades unless they are known to be 304L or 316L stainless steel because of the known interactions of lower carbon containing steels and glass in solution. [16] If a small laboratory grinding mill such as a Tekmar® Grinding Mill is used, ensure that the blade is ~~with a~~ tungsten carbide and not mild steel. Because of the brittle nature of the tungsten carbide blades glass samples should be ~1 to 1.5 cm before using the grinding mill. The sample basket of laboratory grinding mills can also be made of steel. If the sample basket becomes dull due to erosion of the steel, replace the sample basket.

21.5.2 Clean brass or stainless steel sieves, catch pan and lid before and after every use.[26]

21.5.3 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 placing the fragments on the largest mesh chosen sieve. The smallest mesh chosen should be under the largest mesh sieve with a catch pan below. The recommended mesh sizes are 100 mesh (149 micron) and 200 mesh (74 micron).

21.5.4 Place the lid on the nest of sieves and sieve mechanically or manually for approximately 5 minutes.

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

21.5.6 Tap the smallest mesh sieve lightly over colored paper. For example if the glass is light colored tap the sieve on dark paper. For dark glass, e.g. black simulated nuclear waste glass, tap the sieve on white paper. If a significant amount of powder appears on paper, repeat steps 21.5.4 through 21.5.6 until a minimal amount of glass powder passes through the smallest mesh sieve.

21.5.7 Transfer the fraction of the sieved glass to be used for testing into a clean container labeled with the sample identification. The date and the name of the person preparing the sample should also appear on the container.

21.5.8 If additional material is needed, recrush the fragments of glass lying on top of the largest sieve screen or repeat step 21.5.1. When new glass fragments have been prepared repeat steps 21.5.3 through 21.5.4.

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

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[26] *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 and/or high temperatures as the brass mesh has a protective film to inhibit corrosion.*

21.6 *Washing the Sized Glass* -

21.6.1 Place sieved glass in a clean glass beaker which will hold about 2.5 times the sample volume. For example if 15-20 grams of sieved glass is used, a 50 mL glass beaker should be used.

21.6.2 Forcibly add of ambient temperature ASTM Type I water from a squirt bottle to the glass. The volume of water added should be about twice the sample volume. For example if 15-20 grams of glass is used add 25-30 mL of ASTM Type I water. During water addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

21.6.3 Allow the glass-water mixture to settle ~15 seconds, then decant off the water.

21.6.4 Repeat steps 21.6.2 to 21.6.3.

21.6.5 Repeat step 21.6.2.

21.6.6 Prepare the ultrasonic cleaner by filling with water to ~1 cm. Place the beaker from step 21.6.5 in the ultrasonic cleaner for 2 minutes. After removing the beaker from the cleaner, decant the water from the beaker and discard.

21.6.7 Repeat 21.6.6.

21.6.8 Forcibly add absolute alcohol from a squirt bottle to the glass. The volume of alcohol added should be about twice the sample volume. For example, if 15-20 grams of glass is used add 25-30 mL of absolute alcohol. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

21.6.9 Place the beaker from step 21.6.8 in the ultrasonic cleaner for 2 minutes. After the 2 minutes decant the alcohol from the beaker.

21.6.10 Repeat step 21.6.9 two more times.

21.6.11 Put the beaker full of cleaned glass in a 90±2°C oven overnight to dry. Store glass in a clean, sealed and labeled container in a dessicator until use. Use within 3 months. If the glasses have not been stored in a dessicator or they have been stored in a dessicator for over 3 months they must be redried at 90±2°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.

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

22. *Preparation of Reference Glass for PCT Test Method B*

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, 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.

22.1 *Reference Glass Handling* - same as 21.1.

22.2 *Reference Glass Sample Size* - same as 21.3.

22.3 *Number of Reference Glass Replicates* - same as 21.4.

22.4 *Crushing, Sieving, and Washing of Reference Glass* - same as Sections 21.5 and 21.6.

23. *Procedure - PCT Test Method B*

23.1 *Number of Sample Replicates* - All tests for each glass should be carried out at least in triplicate (see Section 21.4).

23.2 *Number of Reference Glass Replicates* - A standard glass test shall be run at least in triplicate as part of each "set" of samples.

23.2.1 A "set" of samples is considered to be those which are tested simultaneously in the same oven.

23.3 *Number of Vessel Blanks* - A blank is considered to be a cleaned test vessel which 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.

23.3.1 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 I.

23.4 *Leaching Method* - Each sample, standard, and blank shall be tested according to the following method:

23.4.1 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. Other leachants can be used, including but not limited to simulated groundwater, actual groundwater, seawater, brine, pH buffers. 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.

23.4.2 Calibrate the pH meter. Determine the pH of an aliquot of the leachant. Put the leachant in a sealed cleaned vessel for transport. Keep the water 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 ASTM D1125). 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 I. Initial all measurement entries on the sheet. Discard the aliquot of leachant used for the pH measurement.

23.4.3 Standardize the balance according to Section 7.2.2. It is recommended that a user's log of the balance standardization be kept. Record the balance identification number and the annual calibration date.

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

23.4.5 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.

23.4.6 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 the same amount of water but no sample.

23.4.7 The "set" of samples including the reference glass vessels, and blanks should be placed immediately into the an 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 7 days. Record the date and time (d:h:min) on a sample log sheet and on the recording device which continuously monitors the oven temperature.

23.4.8 Leave sample vessels in oven at test temperature between 4-16 hours 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 oven for the remainder of the test duration. This is especially important when using PFA Teflon® vessels. Note which lids required additional tightening on a sample log sheet.

23.4.9 The testing period shall be controlled to within plus or minus 1% of the total time period of the test. At the conclusion of the test remove the leach container from the oven and allow the container to cool to room temperature. 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 23.4.11 to 23.4.14 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 which continuously monitors oven temperature.

23.4.10 Check the balance calibration according to Section 7.22. 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 1% of the original leachant mass, abandon 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 1% of the original leachant mass, abandon the triplicate tests and the test specimens. Repeat the test in triplicate starting with new test specimens.

23.4.11 Calibrate the pH meter (see Section 7.2.3 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.

23.4.12 Draw a sufficient amount of the remaining leachant through a 0.45 μm filter into a clean disposable syringe.[23,9] Note that more than one syringe filter may be necessary per sample. Remove the filter and transfer the contents of the syringe into a clean specimen bottle[24] for cation analysis. For poorly durable glasses the solution will need to be diluted with ASTM Type I water before acidification (Section 23.4.13) in order to prevent gellation of the leachate when acidified. Leachates with pH values above 11 should be diluted with ASTM Type I water as a precaution. If optional anion analyses are desired draw another filtered aliquot of the sample and transfer the contents of the syringe into a cleanspecimen bottle. Samples for anion analysis are not acidified (Section 23.4.14).

23.4.13 The solution must always be analyzed for Na, B, Si, Li, and K. Acidify the aliquots drawn for cation analysis with concentrated ultra high purity HNO_3 equal to 1% of the aliquot volume. [27] Perform cation analyses and include acidified multielement solution standards.

23.4.14 Anion analysis is optional but is strongly recommended. Send the aliquot drawn for anion analysis but do not acidify the leachants. Perform anion analysis and include unacidified multielement solution standards. Note the analytic service identification number on a sample log along with the analysis requested.

23.4.15 Measure cation and anion concentrations of glass leachates, standard glass leachates, blanks, and multielement solution standards (consult ASTM C 1109 and D 4327). The short-term

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[27] *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*

precision of these analytic methods at concentrations at least 100 times the detection limit range from 0.3 to 2% relative standard deviation.[28] Precision degrades with decreasing concentration to approximately 25% relative standard deviation at approximately two times the detection limit. [29]The detection limits for each analysis should accompany the reported results.

23.4.16 Analysis of the solids on the filter or the remaining solid glass sample is optional. If solids analysis are not desired, the filter and solid glass sample may be discarded. If solids analysis is desired, record the appearance of the specimen powder, e.g., visible changes in color, agglomeration, and gelatinization. Wash the specimen powder from the leach container with pure water onto a clean watch glass and dry at not > 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.

24. Calculation and Reporting for PCT Test Methods A and B

24.1 *Use of Multielement Standard* - Calculate the mean of the triplicate analyses and standard deviation of the analytic results of the multielement standard. If the average values agree within 10% of the standard values then the analytic results of the study are considered acceptable.

24.2 *Use of Blanks* - Blanks are used to check if sources of contamination are present due to incorrect vessel cleaning or contamination. Corrections of up to 1% of any contaminant element are subtracted from the leachate concentrations for all samples using the data from the replicate blanks from the same batch of cleaned vessels. If corrections of >1% of any contaminant element occur, all the tests performed using vessels from that vessel cleaning batch must be repeated. Leachate concentrations, especially those for the major soluble elements in the glass (Li, B, Na, K, and Si) are measured for the replicate blanks.

24.3 *Calculations of Leachate Concentrations* - Leachate concentrations, especially those for the major soluble elements in the glass (Li, B, Na, K, and Si) are calculated for the test glasses and the standard glass.

24.3.1 Calculate the final leachate volume (V_f) for for each test including blanks. The amount of water loss is equal to the weight loss that occurred during heating. Leachate loses of greater than 5% of the

[28] *ASTM C1109*

initial volume invalidate Test Method A data, while leachate loses of greater than 1% of the initial volume invalidate Test Method B data (see Sections 20.4.9 and 23.4.10).

24.3.2 Calculate the average blank concentration in appropriate units for each element, i, by dividing its respective concentrations by the number of valid tests.

24.3.3 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, 1% of the leachate concentrations.

24.3.4 Calculate the average concentration for each element by dividing the sum of the corrected concentrations by the number of tests performed.

24.3.5 Calculate the standard deviation for the corrected leachate concentrations for various elements.

24.4 *Reporting and Deviations*

24.4.1 Report all results as the concentration of the elements in solution or as the concentration of the elements in solution normalized by the amount of that element present in the glass.

24.4.2 All data should be recorded in a retrievable manner. Deviations from the test method and the expected effect on the results should be discussed.

25. Precision and Bias for PCT Test Methods A and B[20]

25.1 *Precision:*

25.1.1 The data used to generate the measures of precision for PCT Test Method B is the result of intra- and inter-laboratory round robins. 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 ASTM Practice E691. These measures are designated as follows:

[20] *Precision and bias cited from references 7 and 8 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 but sample bias remains the same (C.M. Jantzen and N.E. Bibler, in preparation)*

Repeatability: the standard deviation for within-laboratory determinations.

Reproducibility: the standard deviation for between-laboratory determinations.

25.1.2 *PCT Method A Within - Laboratory Precision:* There is no round robin data to support a statement concerning repeatability and reproducibility. Data from two seperate laboratories has been used to determine the within laboratory precision for remote radioactive operation (Sections 25.1.2.1 through 25.1.2.2).

25.1.2.1 *Radioactive Glass 200R* (a radioactive borosilicate glass containing U-235 neutron fission and activation products). Within-lab standard deviations for B were 2.2 and 5.3% using unwashed 100-200 mesh glass in ASTM Type I water for 7 days (Data in Table III[30]). For Si the within lab standard deviations were 0.7 and 1.1%.

25.1.2.2 *Radioactive Glass 165/42* (a radioactive borosilicate waste glass containing U-235 neutron fission and activation products). Within-lab standard deviations for B were 2.9 and 3.5% using unwashed 100-200 mesh glass in ASTM TYpe I water for 7 days (Data in Table IV [31]). For Si the standard deviation for both laboratories was 3.2%.

25.1.3 *PCT Method A Between - Laboratory Precision:* There is no round robin data to support a statement concerning repeatability and reproducibility. Data from two seperate laboratories has been used to determine the between laboratory precision for remote radioactive operation (Sections 25.1.3.1 through 25.1.3.2).

25.1.3.1 *Radioactive Glass 200R* (a radioactive borosilicate glass containing U-235 neutron fission and activation products). Between-lab relative standard deviations for B and Si were 13% and 11%, respectively, using unwashed 100-200 mesh glass in ASTM Type I water for 7 days (Data in Table III[31]).

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[30] *N.E. Bibler and J.K. Bates, "Product Consistency Leach Tests of Savannah River Site Radioactive Waste Glasses, "Scientific Basis for Nuclear Waste Management , XIII," V.M. Oversby and P.W. Brown (Eds.), Materials Research Society, Pittsburgh, PA, 327-338 (1990),*

25.1.3.2 *Radioactive Glass 165/42* (a radioactive borosilicate waste glass containing U-235 neutron fission and activation products). Between-lab relative standard deviation for B and Si was 14% using unwashed 100-200 mesh glass in ASTM Type I water for 7 days (Data in Table IV [31]).

25.1.4 *PCT Method B Within-Laboratory Precision:*

25.1.4.1 *Approved Reference Material (ARM-1,* a simulated borosilicate nuclear waste glass). Within-lab relative standard deviation for B was 2.3% and for Si was 1.8% using unwashed 100-200 mesh glass in ASTM Type I water for 7 days.[9]

25.1.5 *PCT Method B Between - Laboratory Precision:*

25.1.5.1 *Approved Reference Material (ARM-1,* a simulated borosilicate nuclear waste glass). Between-laboratory relative standard deviation (including within laboratory and between laboratory %RSD) for B was 12% and for Si was 10.2% for laboratories with varying analytic capabilities. Unwashed 100-200 mesh glass in ASTM Type I water for 7 days was used in the intra-laboratory comparison (Data in Table C.2 [8]).

25.1.5.2 -*NIST Reference Glass SRM 623* (a borosilicate glass). Between-laboratory standard deviation (including within laboratory and between laboratory %RSD) for B was 19.8% and for Si was 18.3% for laboratories with varying analytic capabilities. Unwashed 100-200 mesh glass in ASTM Type I water for 7 days was used in the intra-laboratory comparison (Data in Table C.2[8]).

25.2 *Bias:*

25.2.1 For both PCT Method A and B the average corrected leachate concentrations for the standard glass allow assessment of long term bias or variability of the test, e.g. how reproducible the experimental variables such as oven temperature, sieving, leachate analyses, etc. are over time. Use of a standard glass provides the basis for both within-laboratory and between-laboratory data comparisons.

Appendix I 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 # _____ Batch Cleaning #_____
2. Vessel Cleaned on _____ by _____
3. 1st Vessel Blank # _____ Batch Cleaning #_____
4. 2nd Vessel Blank # _____ Batch Cleaning #_____
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/temp (°C) of leachant	_____	_____	_____	_____	_____
4. pH/temp (°C) of buffer solutions	_____	_____	_____	_____	N/A
5. pH meter ID #	_____	_____	_____	_____	N/A
6. wt of empty vessel (gms)	_____	_____	N/A	N/A	N/A
7. wt of vessel + lid +sample(gms)	_____	_____	N/A	N/A	N/A
8. wt of sample (Item 7-6 gms)	_____	_____	N/A	N/A	N/A
9. mL of solution (Item 8 x10)	_____	_____	N/A	N/A	N/A
10. wt of vesel + sample + lid + solution	_____	_____	_____	_____	_____*
11. test and temp **PCT Version 5.0 @ 90°C**				_____	_____
12. date, hour, min test started and ended	_____	_____	_____	_____	_____**
13. vessel lid re-tightened (Y/N)	_____	_____	N/A	N/A	N/A

* **if greater than 5% of initial value for PCT Test Method A data is not useable: if greater than 1% of initial value for PCT Test Method B data is not useable:**

** **If greater than 1% of total time period then data is not useable**

Leachate Analyses

Acidified with	_____	on	_____	by	_____
Diluted with	_____	on	_____	by	_____
Dilution factor	_____	on	_____	by	_____

Analytic Service ID's

Undiluted Cation Analysis #	_____	for	_____
Diluted Cation Analysis #	_____	for	_____
Corresponding Blank ID # (Optional)	_____	for	_____
Undiluted Anion Analysis #	_____	for	_____
Diluted Anion Analysis #	_____	for	_____
Solids Analysis #	_____	for	_____

REMARKS/DEVIATIONS/COMMENTS:

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