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Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)[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~~ glasses, phase separated glasses, devitrified glasses, glass ceramics, and/or multiphase glass ceramic waste forms hereafter collectively referred to as "glass waste forms" by measuring the concentrations of the chemical species released ~~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 glass waste ~~glasses~~forms have been consistently controlled during production. This test method is applicable to radioactive and simulated glass waste ~~glasses.~~ forms as defined above.

1.1.2 Test Method B is a ~~crushed glass~~ durability test that allows testing ~~of waste glasses~~ at ~~varying~~various test durations, test temperatures, mesh size, mass of ~~glass,~~sample, leachant volume, and leachant ~~types.~~compositions. This test method is static and can be conducted in stainless steel or PFA TFE-fluorocarbon vessels, or both. Test Method B can specifically be used to evaluate the relative chemical durability characteristics of homogeneous ~~or~~ glasses, phase separated glasses, devitrified glasses, ~~or both.~~ glass ceramics, and/or multiphase glass ceramic waste forms. This test method is applicable to radioactive (nuclear) and mixed, hazardous, and simulated waste ~~glasses.~~ forms as defined above. Test Method B cannot be used as a consistency test for production of high level radioactive glass waste ~~glass.~~ forms.

1.2 These test methods must be performed in accordance with all quality assurance requirements for acceptance of the data.
~~1.3~~

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 92 Test Methods for Sieve Analysis and Water Content of Refractory Materials
C 162 Terminology of Glass and Glass Products
C 169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass
C 225 Test Methods for Resistance of Glass Containers to Chemical Attack
C 371 Test Method for Wire-Cloth Sieve Analysis of Nonplastic Ceramic Powders
C 429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture
C 693 Test Method for Density of Glass by Buoyancy
C 1109 ~~Test Method~~ Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission ~~Spectrometry~~ Spectroscopy
C 1174 Practice for Prediction of the Long-Term Behavior of Materials, Including Waste Forms, Used in Engineered Barrier Systems (EBS) for Geological Disposal of High~~–~~-Level Radioactive Waste

~~[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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[1] These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.13 on Spent Fuel and High Level Waste.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* ~~, Vol 15.01.~~ volume information, refer to the standard's Document Summary page on the ASTM website.

~~C1317Practice for Dissolution of Silicate or Acid Resistant Matrix Samples[4]~~
~~C1342Practice for Flux Fusion Sample Dissolution[4]~~ 1463 Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis
~~D~~C 1125~~Test Methods for Electrical Conductivity and Resistivity of Water~~ Test Method for Penetration Index of Asbestos
D 1129 Terminology Relating to Water
D 1193 Specification for Reagent Water
D 1293 Test Methods for pH of Water
D 4327 Test Method for Anions in Water by Chemically Suppressed Ion Chromatography
E 7 Terminology Relating to Metallography
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E 456 Terminology Relating to Quality and Statistics
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E 1402 Terminology Relating to Sampling

3. Terminology

3.1 *Definitions:*

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

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

3.1.3 *ASTM Type I water*—purified water with a maximum total matter content including soluble silica of 0.1 g/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~~waste form to the release of its constituents to an aqueous solution under the specific conditions of this test.

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

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

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

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

3.1.8 ~~*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); a noncrystalline solid or an amorphous solid.[3]

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

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

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

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

~~3.1.11~~

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

~~3.1.12~~

3.1.14 *mixed waste*—waste containing both radioactive and hazardous components regulated by the Atomic Energy Act (AEA) **(1)**[4] and the Resource Conservation and Recovery Act (RCRA), **(2)** respectively; the term "radioactive component" refers only to the actual radionuclides dispersed or suspended in the waste substance **(3)** .

~~3.1.13~~

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

~~3.1.14~~

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

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

~~3.1.15~~

3.1.18 *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~~

~~[3] Annual Book of ASTM Standards, Vol 15.02.~~
[3] Varshneya, A. K., "Fundamentals of Inorganic Glasses," Academic Press, Boston, MA (1994).
~~[4] Annual Book of ASTM Standards, Vol 12.01.~~
[4] The boldface numbers in parentheses refer to the list of references at the end of these test methods.

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

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

~~3.1.17~~3.1.21 *radioactivity*—spontaneous nuclear disintegration with emission of corpuscular or electromagnetic radiation, or both (consult Terminology D 1129).

~~3.1.18~~

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

3.1.23 *sensitization—in austenitic steels such as Types 304 and 316*, the precipitation of chromium carbide at the grain boundaries in a temperature range of 400–900°C (modified from Terminology E 7).

~~3.1.19.1~~

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

~~3.1.20~~

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

~~3.1.21~~

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

~~3.1.22~~

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

~~3.1.23~~*)*

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

~~3.1.24~~

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

~~3.1.25~~

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

~~3.1.26~~

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

4. Summary of Test Methods

4.1 Test Method A is the Product Consistency Test (PCT-A), which was developed specifically to ~~test~~measure the chemical durability of radioactive glass waste ~~glasses~~forms as defined in 1.1 during production (Table 1) **(5)**. It can also be used to ~~test~~ measure the chemical durability of hazardous, mixed, and various simulated glass waste ~~glasses.~~ forms as defined in 1.1. The test method is easily reproducible, can be performed remotely on highly radioactive samples and can yield results rapidly. The glass waste form does not need to be annealed prior to testing. In this test method the glass waste form is crushed and sieved to U.S. Standard ASTM − 100 to + 200 mesh (0.149–0.074 mm), the particles are cleaned of adhering fines, and an amount of sized and cleaned glass waste form that is greater than or equal to 1 g is placed in a Type 304L stainless steel vessel. An amount of ASTM Type I water equal to 10 ~~cc/g~~ ± 0.5 cm^3/g of sample mass (m_{solid})[5] is added and the vessel is sealed. The vessel is placed in a constant temperature device at 90 ± 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~~measure the chemical durability of radioactive, mixed, or simulated glass waste ~~glasses~~forms **(5)**. The test method is easily reproducible, can be performed remotely if necessary, and can yield results rapidly. The glass waste form does not need to be annealed prior to testing. In this test method the glass waste form is crushed and sieved to U.S. Standard ASTM − 100 to + 200 mesh (0.149–0.074 mm) or to the size range of interest as long as the glass waste form particles are less than U.S. Standard ASTM 40 mesh (0.420 mm). The particles are cleaned of adhering fines (see Note 1), and an amount of sized and cleaned glass waste form greater than or equal to 1 g is placed

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

[5] If waste forms of different densities are being compared then the leachate results from the test must be compared using the calculation in 25.2.4 which accounts for density differences in the SA/V term in the denominator which adjusts the leachate results for sample density (see calculation in Appendix X1).

TABLE 1 Summary of Test Methods A and B

	Test Method A	Test Method B
~~Type of Glass~~	~~Radioactive Mixed Simulated, Hazardous~~	~~Radioactive Mixed Simulated, Hazardous~~
Type of Waste Form	Radioactive Mixed Simulated, Hazardous	Radioactive Mixed Simulated, Hazardous
Usage	During production for rapid analysis and for waste compliance **(6)**	Scoping tests; Crystallization studies (see Note 1); Comparative waste form evaluation
~~Test Vessel~~	~~Unsensitized Type 304L stainless steel; vessels rated to> 0.5 MPa (see Section 9)~~	~~Unsensitized Type 304L stainless steel or PFA TFE fluorocarbon[9] vessels rated to >0.5 MPa (see 9)~~
Test Vessel	Unsensitized Type 304L stainless steel; vessels rated to> 0.5 MPa (see Section 9)	Unsensitized Type 304L stainless steel or PFA TFE-fluorocarbon[9] vessels rated to >0.5 MPa (see Section 9)
~~Test Duration~~	~~7 days ± 3.4 h~~	~~7 days ± 3.4 h or varying times~~
Test Duration	7 days ± 2%	7 days ± 2% or varying times
Leachant	ASTM Type I water	ASTM Type I water or other solutions
Condition	Static	Static
~~Sample Mass~~	~~≥1 g~~	~~≥1 g~~
Minimum Sample Mass	≥1 g	≥1 g
Particle Size	U.S. Standard ASTM – 100 to + 200 mesh (0.149 to 0.074 mm)	U.S. Standard ASTM – 100 to + 200 mesh (0.149 to 0.074 mm) or other sizes which are <40 mesh (0.420 mm)
~~Leachant Volume~~	~~10 cc/gram of sample mass~~	~~10 cc/gram of sample mass or variable volume/sample mass~~
Leachant Volume	10 ± 0.5 cm^3/gram of sample mass	10 ± 0.5 cm^3/gram of sample mass or other volume/sample mass
Temperature	90 ± 2°C	90 ± 2°C or other temperatures provided that any observed changes in reaction mechanism are noted
Atmosphere	Air	Air or CO_2 free air (optional) (see Section 10)
Type of System	Closed to transport	Open to transport in PFA TFE-fluorocarbon; Closed to transport in stainless steel

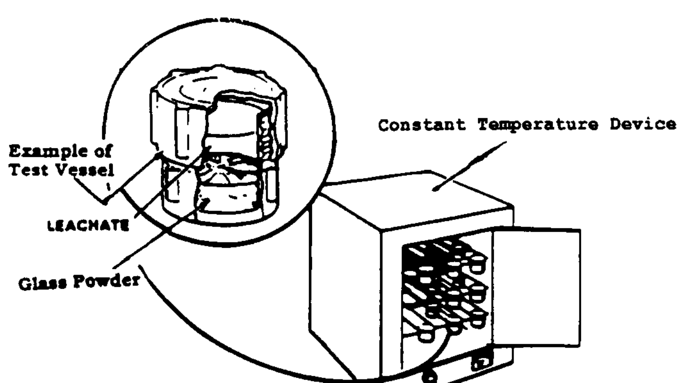

FIG. 1 Schematic of Test Apparatus

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~~

± 0.5 cm^3/g of sample mass (m_{solid})[5] is added and the vessel is sealed. Other ratios of solution volume to sample mass are allowed and other leachants are allowed. The vessel is placed in a constant temperature device at 90 ± 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~~a constant temperature device 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~~ 1—Devitrified glasses, glass ceramics, and multiphase glass ceramic waste forms containing soluble secondary phases require special handling procedures (see 19.6.1 and 22.6.1).

5. Significance and Use

5.1 These test methods provide data useful for evaluating the chemical durability (see 3.1.4) of glass waste forms as measured by elemental release. Accordingly, it may be applicable throughout manufacturing, research, and development.

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

5.1.2 Test Method B can specifically be used to measure the chemical durability of glass waste forms under various leaching conditions, for example, varying test durations, test temperatures, ratio of ~~glass surface~~sample-surface area (S) to leachant volume (V) (see Appendix X1), and leachant types (see Table 1). Data from this test may form part of the larger body of data that are necessary in the logical approach to long-term prediction of waste form behavior (see Practice C 1174).

6. Apparatus

6.1 *Test Vessels for Test Method A*— The production test method requires the use of unsensitized Type 304L stainless steel leach vessels of >20 mL capacity designed to take internal pressures of > 0.5 MPa without leaking (see Sections 10 and 11).

6.1.1 The stainless steel vessels require a gasket material in order to remain sealed. TFE-fluorocarbon gaskets, available commercially, are acceptable for test durations of less than 28 days since TFE-fluorocarbon is chemically inert and exposure to radiation doses up to 1×10^5 rad of beta or gamma radiation have been shown **(7)** not to damage TFE-fluorocarbon. If higher radiation doses are present, degradation of the TFE-fluorocarbon gasket can compromise the seal or contaminate the leachate with fluoride, or both, as F^- and HF **(8)**. ~~In~~For high radiation doses, special gaskets fabricated from metals such as copper, gold, lead or indium are recommended. High radiation doses will not be experienced with hazardous, mixed, or simulated glass waste ~~glasses.~~ forms.

6.2 *Test Vessels for Method B*—Test Method B allows for the use of either unsensitized Type 304L stainless steel or PFA TFE-fluorocarbon leach vessels of >20 mL capacity designed to take internal pressures of >0.5 MPa without leaking (see Section 10) **(6)**.

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

6.2.2 High radiation ~~doses~~fields (>1×10^5 rad of beta or gamma radiation) will not generally be ~~experienced with~~generated by hazardous, mixed or simulated nuclear waste ~~glasses.~~ glass forms. PFA TFE-fluorocarbon vessels, available commercially, can be used in the absence of high radiation ~~doses~~ fields[6] 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 ove~~n~~r-temperature control.

6.4 *Conventional Oven*~~—Optional laboratory or drying ovens,~~—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~~recording frequency should be 0.5 h.

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

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

6.8 *Crushing Device*—Any mechanical or manual crushing device that will avoid iron (mild steel) contamination in the crushed ~~glass~~waste form specimen **(10)** . Crushing and grinding devices made of tungsten carbide, agate, sapphire, stainless steel, or dense alumina are acceptable.

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

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

6.9 *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.[7]

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

6.14 *pH meter*—pH meter with an accuracy of ±0.1 pH units.

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

6.16 *Ultrasonic Cleaner*.

6.17 *Analytic Equipment*—Equipment for measuring anion and cation content of the leachates and anion content of dilute solutions, for example, inductively coupled plasma-atomic emission spectrometry (see Test Methods C 1109 or Ref **11**, or both), atomic absorption spectrometry, ion chromatography (consult Test Method D 4327 or Ref **11**, or both), or ion selective electrodes, or a combination thereof.

7. Standards

7.1 ~~*Reference Glass*—A reference glass (see Ref~~ **Reference Waste Form**—A reference waste form (see Ref **12** for example) of choice, similar in composition to the glass waste form being tested, is to be tested in triplicate along with each batch of glass waste forms tested (see Ref **13, 44**). The reference ~~glass~~waste form composition should be traceable to NIST, or to a comparable source.

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

7.3 *pH Buffers*—commercial pH buffers or pH buffers made to the specifications given in Test Method D 1293 that bracket the measured pH range of the leachant and leachate. All commercial buffer solutions should be traceable to NIST, or a comparable source, and have a certified shelf life. Keep all the reference buffer solutions well sealed and replace at the expiration of shelf life, or sooner if a visible change is observed (see Test Method D 1293).

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

8. Reagents and Standards

8.1 *ACS Reagent Grade Acids*—Reagent grade nitric acid (HNO_3) and hydrofluoric acid (HF) for cleaning leach vessels.[8]

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

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

8.4 *Solvents*~~—Ethanol 95% pure and reagent grade acetone.~~ —Absolute 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~~18 MΩ·cm at 25°C (see Specification D 1193).

8.5.1 The source water shall be purified, then passed through a deionizer cartridge packed with a mixed bed of nuclear-grade resin,[9] then through a cellulose ester membrane having openings not exceeding 0.45 µm.[10]

8.5.2 Pass the purified water through an in-line conductivity cell to verify its purity. Alternatively, the water can be measured for all anions and cations to verify that there is less than a total dissolved solid content including soluble silica of 0.1 g/m^3(see Specification D 1193 and Terminology D 1129).

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

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

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

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

~~[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.~~

[9] A nuclear-grade resin mixture of the strong acid cation exchanger in the hydrogen form and the strong base anion exchanger in the hydroxide form with a one-to-one cation to anion equivalence ration, such as that available from the Millipore Corp., Bedford, MA 01730; Barnstead Co., 225 Rivermoor St., Boston, MA 02131; Illinois Water Treatment Co., 854 Cedar St., Rockford, IL 61105; or Vaponics, Inc., 200 Cordage Park, Plymouth, MA 02360, is suitable. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

~~[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.~~

[10] An in-line filter such as those made by the Millipore Corp., Bedford, MA 01730; Gelman Instrument Co., 600 S. Wagner Rd., Ann Arbor, MI 48106; and Schleicher and Schuell, Inc., 540 Washington St., Keene, NH 10003, has been found to be satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

8.6 *Other Leachants*—Test Method B allows for the use of other leachants such as simulated or real groundwaters, brine, seawater, pH buffers, and others. The simulated solutions should be made from ACS reagent grade chemicals.[8] All leachants should be chemically analyzed to verify their composition before durability testing begins. All leachants should have a specified shelf life.

9. Hazards

9.1 All appropriate precautions for operation of pressurized equipment must be taken. To ensure safe operation, the leach containerstest vessels should be designed to withstand the vapor pressure of water at the test temperature with an appropriate safety factor. The thermal expansion of water must be taken into account when filling the leach containers. For example,Specifically, between 4°C and 100°C, water expands by 4 volume %. Overfilling, for example, filling a 60 mL vessel to 55 mL, may lead to pressures inside the container that exceed the design limits and could lead to the failure of one or more parts of the vessel.

10. Choice of Test Vessel

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

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

10.1.2 It is recommended that 22 mL vessels[11] be used for the radioactive production application in Test Method A to minimize the amount of radioactive sample being handled.

10.2 *PFA TFE-fluorocarbon Vessels*—PFA TFE-fluorocarbon vessels may be used in Test Method B. PFA TFE-fluorocarbon vessels can be used for Test Method B for short-term chemical durability testing with mixed or simulated nuclear glass waste glasses.forms. The use of PFA TFE-fluorocarbon vessels is acceptable for test durations of ≤28 days. Longer test durations are also acceptable only if it can be demonstrated that the vessel interactions do not affect the glass waste form reactivity. The user should ensure that new PFA TFE-fluorocarbon vessels are free from fluoride which is present as a free surface fluoride from vessel fabrication (see Section 16).

10.2.1 PFA TFE-fluorocarbon vessels are for "open system" applications where the influx of 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.

10.2.2PFA 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 1×10

10.2.2 PFA TFE-fluorocarbon vessels cannot be used in Test Method A and it is recommended that PFA TFE-fluorocarbon vessels not be used in Test Method B for radioactive glass waste forms[6]. The use of PFA TFE-fluorocarbon vessels for radiation doses >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 rad causes degradation of the PFA TFE-fluorocarbon, and subsequent release of F^- and HF byinto the test solution. The presence of HF in the solution may change the rate of degradation of the glass waste form due to the acidic conditions and F^- ions that attack the glass waste form **(7, 8)** .

11. Identification of Vessels and Vessel Cleaning History

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

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

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

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

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

[11] Vessels from Parr Instrument Co., 211 53rd St., Moline, IL 61265, have been found satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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

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

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

12.1.2 Clean the vessels and lids ultrasonically in ~~95%~~95% absolute ethanol for approximately 5 min.

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

12.1.4 Submerge the vessels and lids in 0.16 *M* 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 absolute ethanol. Recheck the integrity of the gasket and discard if damaged.

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

13.1.3 Clean each gasket ultrasonically in ~~95%~~95% absolute ethanol for approximately 10 min.

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

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

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

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

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

14.1 When stainless steel vessels are reused subsequent to their use with radioactive specimens, residual contamination may be present. The vessels shall be cleaned before reuse by cleaning with 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~~ any remaining glass waste form sample from previous testing by rinsing the vessel and lid with ASTM Type I water. Fill the vessel 80 to 90% full with 0.16*M* HNO_3(1 weight % HNO_3). Reseal the vessel and place in 90 ± 10°C oven for a minimum of 16 h to dissolve (acid strip) any radionuclides adhering to the interior of the vessel.

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

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

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

14.1.5 Remove vessels from oven, take one aliquot of water from each vessel and measure the pH (see Test Methods 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 form testing follow 14.1.1 to 14.1.2. For vessels used for non-radioactive glass waste form testing remove ~~all~~ any remaining glass waste form from previous testing by rinsing the vessel and lid with ASTM Type I water.

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

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

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

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

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

15.1.7 Remove vessels from oven, take one aliquot of water from each vessel and measure the pH (see Test Methods 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 µg/mL, repeat steps 16.1.1 to 16.1.12.

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

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

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

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

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

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

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

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

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

17.1.6 Remove vessels from oven, take an aliquot of water from each vessel and measure the pH (see Test Methods D 1293).

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

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

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

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

18. Calibration

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

18.2 *Calibration and Standardization Schedule*:

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

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

18.2.3 *pH meter*—Standardize the pH meter before each use and after completion of all samples with commercial buffer solutions that bracket the solution pH values being measured. Standardize the pH meter at the same temperature as the leachate solutions being measured. If only an occasional pH determination is made, standardize the assembly each time it is used. In a long series of measurements, supplemental interim checks at regular intervals are recommended. In as much as commercially available pH assemblies exhibit different degrees of measurement stability, conduct these checks at intervals of 30 min, unless it is ascertained that less frequent checking is satisfactory to ensure the performance described in Test Methods D 1293.

18.2.4 *Water Purification System*—Calibrate at least annually following the manufacturer's instructions. Standardize before every use with the 10 MΩ·cm at 25°C resistivity calibration cell on the water purification system (see Test Methods D 1125).

PRODUCT CONSISTENCY TEST (PCT)—TEST METHOD A

19. Sample Preparation for PCT Test Method A

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

19.2 *Choice of Appropriate Sample*—Samples of glass waste forms may either be fabricated individually or taken from larger samples ~~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 waste form does not have to be annealed. Flush the sample surface with ASTM Type I water to remove potential surface contamination and dry before crushing.

19.3 *Choice of Sample Mass*~~—The reference ratio of leachant volume to sample mass (~~*—Samples must be ≥ 1 g. The reference ratio of leachant volume to sample mass (V_{soln}/m_{solid}) is 10±* ~~*0.1 cc/g.*~~*0.5 cm^3/g. The volume of leachant is constrained by the volume of the* ~~*leach*~~*test vessel chosen and the need to minimize sample size when dealing with highly radioactive glass waste forms. For example, 1.5 g of sample can be tested in 15 mL of leachant contained in a 22 mL steel vessel.* ~~*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 Waste Forms*—If the glass ~~is not fully oxidized (redox sensitive),~~ waste form is redox sensitive, the user is cautioned that grinding the ~~glass~~ waste form in advance of the test may cause the ~~glass~~ waste form to oxidize and may alter the leachate results. Redox sensitive glass waste forms shall be used within two days of grinding (see Test Methods C 169 and Ref **24**). If the sample has dimensions larger than 2 cm, wrap the sample in a clean plastic bag and break it into smaller fragments with a hammer. It may be necessary to use steel crushing devices. Mild steel should be avoided due to the known interactions of mild steel and glass waste forms in solution (**10**). Crushing devices made of Types 304L and 316L stainless steel should be used to minimize these effects. ~~A~~ It is recommended that a representative ~~sample~~amount of the material to be tested shall weigh at least twice the required weight of the amount of ~~glass~~sample needed to perform the PCT in triplicate in order to have enough ~~glass~~sample to test.

19.5.1 Transfer ~~glass~~ waste form fragments into a clean manual or mechanical grinder. Clean the grinder prior to crushing a different ~~type of glass.~~ waste form. Do not use mechanical grinders with steel blades unless they are known to be made of Types 304L or 316L stainless steel because of the known interactions of mild steels and glass waste forms in solution (**10**). If a laboratory size grinding mill is used, ensure that the blade is tungsten carbide and not mild steel. Because of the brittle nature of the tungsten carbide blades, glass waste form samples should be less than 1.0 cm before using the grinding mill. The sample basket of laboratory grinding mills should be made of stainless steel. If the sample basket appears dull due to erosion of the stainless steel, replace the sample basket. Use of mild steel baskets is not allowed (see Section 6.8).