Designation: C 1285 – 02

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

This standard is issued under the fixed designation C 1285; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These product consistency test methods A and B evaluate the chemical durability of homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, and/or multiphase glass ceramic waste forms hereafter collectively referred to as "glass waste forms" by measuring the concentrations of the chemical species released to a test solution.

1.1.1 Test Method A is a seven-day chemical durability test performed at 90 ± 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 forms have been consistently controlled during production. This test method is applicable to radioactive and simulated glass waste forms as defined above.

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

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

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

2. Referenced Documents

2.1 *ASTM Standards:*

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

C 162 Terminology of Glass and Glass Products[3]

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

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

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

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

C 693 Test Method for Density of Glass by Buoyancy

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

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

C 1463 Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis[4]

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

D 1129 Terminology Relating to Water[5]

D 1193 Specification for Reagent Water[5]

D 1293 Test Methods for pH of Water[5]

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

E 7 Terminology Relating to Metallography[6]

E 177 Practice for Use of the Terms Precision and Bias in

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

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

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

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

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

ASTM Test Methods[7]
E 456 Terminology Relating to Quality and Statistics[7]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[7]
E 1402 Terminology Relating to Sampling[7]

3. Terminology

3.1 *Definitions:*

3.1.1 *anneal*—to prevent or remove materials processing 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 waste form to the release of its constituents to an aqueous solution under the specific conditions of this test.

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

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

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

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

3.1.8 *glass*—an inorganic product of fusion that has cooled to a rigid condition without crystallizing (see Terminology C 162); a noncrystalline solid or an amorphous solid.[8]

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

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

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

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

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

3.1.14 *mixed waste*—waste containing both radioactive and hazardous components regulated by the Atomic Energy Act (AEA) **(1)**[9] and the Resource Conservation and Recovery Act (RCRA), **(2)** respectively; the term "radioactive component" refers only to the actual radionuclides dispersed or suspended in the waste substance **(3)**.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

4. Summary of Test Methods

4.1 Test Method A is the Product Consistency Test (PCT-A), which was developed specifically to measure the chemical

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

[8] Varshneya, A. K., "Fundamentals of Inorganic Glasses," Academic Press, Boston, MA (1994).

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

durability of radioactive glass waste forms as defined in 1.1 during production (Table 1) **(5)**. It can also be used to measure the chemical durability of hazardous, mixed, and various simulated glass waste forms as defined in 1.1. The test method is easily reproducible, can be performed remotely on highly radioactive samples and can yield results rapidly. The glass waste form does not need to be annealed prior to testing. In this test method the glass waste form is crushed and sieved to U.S. Standard ASTM − 100 to + 200 mesh (0.149–0.074 mm), the particles are cleaned of adhering fines, and an amount of sized and cleaned glass waste form that is greater than or equal to 1 g is placed in a Type 304L stainless steel vessel. An amount of ASTM Type I water equal to 10 ± 0.5 cm^3/g of sample mass (m_{solid})[10] 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 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.

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

TABLE 1 Summary of Test Methods A and B

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

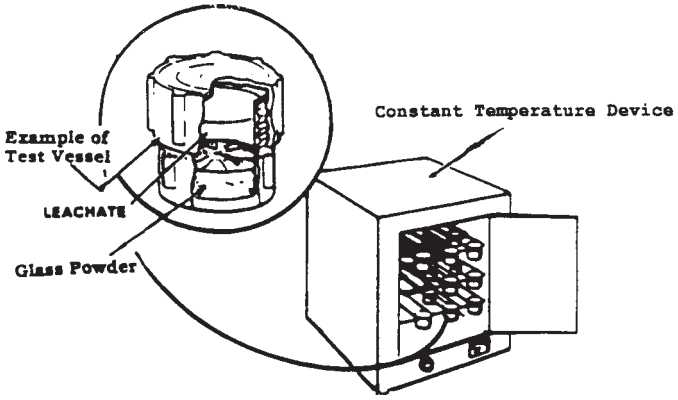

FIG. 1 Schematic of Test Apparatus

4.2 Test Method B is the Product Consistency Test (PCT-B), which was developed to measure the chemical durability of radioactive, mixed, or simulated glass waste forms **(5)**. The test method is easily reproducible, can be performed remotely if necessary, and can yield results rapidly. The glass waste form does not need to be annealed prior to testing. In this test method the glass waste form is crushed and sieved to U.S. Standard ASTM − 100 to + 200 mesh (0.149–0.074 mm) or to the size range of interest as long as the glass waste form particles are less than U.S. Standard ASTM 40 mesh (0.420 mm). The particles are cleaned of adhering fines (see Note 1), and an amount of sized and cleaned glass waste form greater than or equal to 1 g is placed in either a Type 304 L stainless steel vessel or a PFA TFE-fluorocarbon vessel. An amount of ASTM Type I water equal to 10 ± 0.5 cm^3/g of sample mass (m_{solid})[10] 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 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, 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 forms have been consistently controlled during production (see Table 1).