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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 C1285; 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 provide a measure of the chemical durability of homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, multiphase glass ceramic waste forms, or combinations thereof, hereafter collectively referred to as "glass waste forms" by measuring the concentrations of the chemical species released to a test solution under carefully controlled conditions.

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. The stainless steel vessels require a gasket to remain leak-tight.[2] The stainless steel vessels are considered to be "closed system" tests. 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, particle size and masses of glass sample, leachant volumes, and leachant compositions. This test method is static and can be conducted in stainless steel or PFA TFE-fluorocarbon vessels. The stainless steel vessels are considered to be "closed system" while the PFA TFE-fluorocarbon vessels are considered to be "open system" tests. Test Method B can specifically be used to evaluate the relative chemical durability characteristics of homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, or multiphase glass ceramic waste forms, or combinations thereof. This test method is applicable to radioactive (nuclear) and mixed, hazardous, and simulated glass waste forms as defined above. Test Method B cannot be used as a consistency test for production of high level radioactive glass waste forms.

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

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C92 Test Methods for Sieve Analysis and Water Content of Refractory Materials
C162 Terminology of Glass and Glass Products
C169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass
C225 Test Methods for Resistance of Glass Containers to Chemical Attack
C371 Test Method for Wire-Cloth Sieve Analysis of Nonplastic Ceramic Powders
C429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture
C693 Test Method for Density of Glass by Buoyancy
C859 Terminology Relating to Nuclear Materials
C1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy

[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] TFE-fluorocarbon gaskets, available commercially, are acceptable and chemically inert up to radiation doses of 1×10^5 R of beta or gamma radiation which have been shown not to damage TFE-fluorocarbon. If higher radiation doses are anticipated, special gaskets fabricated from metals such as copper, gold, lead, or indium are recommended.

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

C1174 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
C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste
C1463 Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis
C1662 Practice for Measurement of the Glass Dissolution Rate Using the Single-Pass Flow-Through Test Method
D859 Test Method for Silica in Water
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1293 Test Methods for pH of Water
D4327 Test Method for Anions in Water by Suppressed Ion Chromatography
D5956 Guide for Sampling Strategies for Heterogeneous Wastes
E7 Terminology Relating to Metallography
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E456 Terminology Relating to Quality and Statistics
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1402 Guide for Sampling Design

3. Terminology

3.1 *Definitions:*Definitions:

3.1.1 *See Terminology C859 for additional references not listed below.*

3.1.2 *anneal, n*—to prevent or remove materials processing stresses in glass by controlled cooling from a suitable temperature (modified from Terminology C162).

3.1.3 *annealing, n—in glass leach tests*, a controlled cooling process for glass designed to reduce thermal residual stress to an acceptable level, and, in some cases, modify structure. (Terminology C859)

3.1.4 *ASTM Type I water, n*—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 D1193 and Terminology D1129).

3.1.5 *chemical durability, n—in leach tests*, the resistance of a material to alteration, dissolution and release of its constituents, under the specific conditions of the test (Terminology C859).

3.1.6 *closed system, n—in leach tests*, system utilizing a test container that is impervious to material transport (Terminology C859).

3.1.7 *consistently controlled, adj—in high level waste vitrification in the US*, that has been controlled in such a way that its chemical durability is consistent, by comparison with a standard or a target, or by other experiments (proposed Terminology C859).

3.1.8 *devitrified glass, n*—an initially homogeneous or phase separated glass, or both, that has partially crystallized during cooling, heat treatment, or both (Terminology C859).

3.1.9 *glass, n*—an inorganic product of fusion that has cooled to a rigid condition without crystallizing (see Terminologies C162 and C859).

3.1.10 *glass ceramic, n*—a solid material composed of both crystalline and glassy phases (Terminology C859).

3.1.11 *hazardous waste, n*—(1) *in waste management in a broad sense*, any substance or mixture of substances having properties capable of producing adverse effects on the health or safety of a human (see also *RCRA hazardous waste*); (2) *in waste management in the US*, any waste that is "listed" in 40CFR Parts 261.31 -261.33 or exhibits one or more of the characteristics identified in 40CFRParts 261.20 -261.24, is a mixture of hazardous and non-hazardous waste, or is determined to be hazardous waste by the generator (proposed Terminology C859).

3.1.12 *hazardous waste glass, n*—a glass comprised of glass forming additives and hazardous waste.

3.1.13 *homogeneous glass, n*—a glass that is a single amorphous phase; a glass that is not separated into multiple amorphous phases (Terminology C859).

3.1.14 *leachant, n—in leach tests*, general term for the initial solution with which a solid is contacted and into which the solid dissolves or is leached (Terminology C859).

3.1.15 *leachate, n—in leach tests*, general term for the solution resulting from a test in which a solid is contacted by a solution and leaches or dissolves (Terminology C859).

3.1.16 *mixed waste, n—in the US*, waste containing radioactive, source special nuclear, or byproduct material regulated by the Atomic Energy Act (AEA) and hazardous components regulated by the Resource Conservation and Recovery Act (RCRA); the term "radioactive component" refers only to actual radionuclides dispersed or suspended in the waste substance (DOE Order 5400.3) (proposed Terminology C859).

3.1.17 *mixed waste glass, n—in nuclear waste management in the US*, a glass composed of glass-forming additives and mixed waste components (proposed Terminology C859).

3.1.18 *nuclear waste glass, n*—a glass composed of glass forming additives and radioactive waste (proposed Terminology C859).

3.1.19 *open system, n—in leach tests*, a system utilizing a test container through which material transport is possible , for example O_2 or CO_2 diffusion, or both (Terminology C859).

3.1.20 *phase separated glass, n*—a glass composed of more than one amorphous phase (Terminology C859).

3.1.21 *radioactive, adj*—of or exhibiting radioactivity (proposed Terminology C859).

3.1.22 *radioactivity, n*—spontaneous nuclear disintegration with emission of corpuscular or electromagnetic radiation, or both (consult Terminology D1129).

3.1.23 *sample blank, n*—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.

3.1.24 *sensitization, n—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 E7).

3.1.24.1 *Discussion*—This constitutes the greatest single threat to their corrosion resistance (**1**).[4]

3.1.25 *set of samples, n*—samples tested simultaneously in the same oven.

3.1.26 *simulated waste glass, n*—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.

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

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

3.1.29 *unsensitized austenitic steel, n*—stainless steel that is not sensitized (see **sensitization**).

3.1.30 *verify, v*—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*)

3.1.31 *vitrification, n*—the process of fusing waste or simulated waste with glass making chemicals at elevated temperatures to form a waste glass or a simulated waste glass (proposedTerminology C859).

4. Summary of Test Methods

4.1 Test Method A is the Product Consistency Test (PCT-A), which was developed specifically to measure the chemical durability of radioactive glass waste forms as defined in 1.1 during production (Table 1) (**2**). 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 repeatable, 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 isolate the size fraction of U.S. Standard ASTM – 100 to + 200 mesh (0.149–0.074 mm) for use in the test, the particles are cleaned of adhering fines (see Note 1), and a weighed 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 ten times the sample mass (m_{solid})[5] is added so that (V_{soln}/m_{solid}) = 10 ± 0.5 mL/g 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 vessels and even heat distribution (Fig. 1). After seven days ±3.4 h, the vessel is removed from the constant temperature device and cooled to ambient temperature. The pH of an aliquot of the leachate is measured and temperature of the aliquot at the time of the pH measurement is also recorded. The remaining leachate is filtered and the filtrate sent for analysis. Tests with a reference glass are to be conducted in parallel with tests with the glass waste form to verify that the tests were conducted and analyzed properly. The test response provides a measure of the amounts of various glass components that are released to solution under carefully controlled conditions that

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 (**3**)	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

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[5] If waste forms of different densities are being compared then the leachate results from the test must be compared using the calculation in 25.3 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).