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Standard Test Method for Measurement of Glass Dissolution Rate Using Stirred Dilute Reactor Conditions on Monolithic Samples[1]

This standard is issued under the fixed designation C1926; 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 This test method describes a test method in which the dissolution rate of a homogenous silicate glass is measured through corrosion of monolithic samples in stirred dilute conditions.

1.2 Although the test method was designed for simulated nuclear waste glass compositions per Guide C1174, the method is applicable to glass compositions for other applications including, but not limited to, display glass, pharmaceutical glass, bioglass, and container glass compositions.

1.3 Various test solutions can be used at temperatures less than 100 °C. While the durability of the glass can be impacted by dissolving species from the glass, and thus the test can be conducted in dilute conditions or concentrated condition to determine the impact of such species, care must be taken to avoid, acknowledge, or account for the production of alteration layers which may confound the step height measurements.

1.4 The dissolution rate measured by this test is, by design, an average of all corrosion that occurs during the test. In dilute conditions, glass is assumed to dissolve congruently and the dissolution rate is assumed to be constant.

1.5 Tests are carried out via the placement of the monolithic samples in a large well-mixed volume of solution, achieving a high volume to surface area ratio resulting in dilute conditions with agitation of the solution.

1.6 This test method excludes test methods using powdered glass samples, or in which the reactor solution saturates with time. Glass fibers may be used without a mask if the diameter is known to high accuracy before the test.

1.7 Tests may be conducted with ASTM Type I water (see Specification D1193 and Terminology D1129), buffered water or other chemical solutions, simulated or actual groundwaters, biofluids, or other dissolving solutions.

1.8 Tests are conducted with monolithic glass samples with at least a single flat face. Although having two plane-parallel faces is helpful for certain step height measurements, it is not required. The geometric dimensions of the monolith are not required to be known. The reacted monolithic sample is to be analyzed following the reaction to measure a corroded depth to determine dissolution rate.

1.9 Tests may be performed with radioactive samples. However, safety concerns working with radionuclides are not addressed in this test method.

1.10 Data from these tests can be used to determine the value of kinetic rate model parameters needed to predict glass corrosion behavior over long periods of time. For an example, see Practice C1662, section 9.5.

1.11 This test method must be performed in accordance with all quality assurance requirements for acceptance of the data.

1.12 *Units*—The values stated in SI units are regarded as the standard. Any values given in parentheses are for information only.

1.13 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.14 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C693 Test Method for Density of Glass by Buoyancy
C859 Terminology Relating to Nuclear Materials
C1174 Guide for Evaluation of Long-Term Behavior of Materials Used in Engineered Barrier Systems (EBS) for Geological Disposal of High-Level Radioactive Waste

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is 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* volume information, refer to the standard's Document Summary page on the ASTM website.

C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste
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
D7778 Guide for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *chemical durability, n*—the resistance of a glass to dissolution under particular test conditions.

3.1.2 *forward glass dissolution rate, n*—the rate at which glass dissolves into solution at specific values of the temperature and pH in the absence of condensation reactions.

3.1.3 *glass coupon, n*—a mechanically sectioned monolith of glass containing at least one flat surface; the flat surface should be polished to a finish of 3000 nm or better.

3.1.4 *gravimetric, adj*—measured by change in mass.

3.1.5 *intrinsic rate constant, n*—the component of the forward rate constant that depends only on the glass composition.

3.1.6 *reaction vessel, n*—a sealed container made of an inert material containing the contacting solution and glass sample.

3.1.7 *step height, n*—the difference in height between an un-corroded portion of a glass monolith sample and the bulk corroded surface.

3.1.8 *test solution, n*—the solution contacting the glass during the experiment.

3.2 Definitions not listed here can be found in Terminology C859.

4. Summary of Test Method

4.1 Based on Test Method C1220, Practice C1662, and the work of Icenhower and Steefel (2015) (**1**)[3], monolithic glass specimens are contacted by a solution by suspending the glass coupon in a well-agitated reaction vessel at a low surface area to volume ratio, to maintain dilute conditions. A portion of the glass coupon surface is masked with an inert material during the solution exposure. Following the exposure, the inert material is removed from the surface or accounted for in the height measurement. The height difference between the protected, un-corroded surface and the receded corroded surface is then measured. This step height is used to calculate the dissolution rate of the glass by using Eq 1:

$$rate = \frac{h \times p}{t} \tag{1}$$

where:

rate	=	dissolution rate of the glass, g/m^2·d,
h	=	depth of surface recession, m,
p	=	density of the glass (that is, by Test Method C693), g/m^3, and
t	=	experimental duration in days, d.

4.2 The step height measurements are to be collected from samples where the dilution of species dissolving from the glass remain at concentration below which they can impact the dissolution of the glass. In doing so, the forward glass dissolution rate at infinite dilution can be determined.

5. Significance and Use

5.1 This test method provides a description of the design of the Stirred Reactor Coupon Analysis (SRCA) apparatus and identifies aspects of the performance of the SRCA tests and interpretation of the test results that must be addressed by the experimenter to provide confidence in the measured dissolution rate.

5.2 The SRCA methods described in this test method can be used to characterize several aspects of glass corrosion that can be included in mechanistic models of long-term durability of glasses, including nuclear waste glasses.

5.3 Depending on the test parameters investigated, the SRCA results can be used to measure the intrinsic dilute glass dissolution rate, as well as the effects of conditions such as temperature, pH, and solution chemistry on the dissolution rate.

5.4 Due to the scalable nature of the method, it is particularly applicable to studies of the impact of glass composition on dilute-condition corrosion. Models of glass behavior can be parameterized by testing glass composition matrices and establishing quantitative structure-property relationships.

5.5 The step heights present on the corroded sample can be measured by a variety of techniques including profilometry (optical or stylus), atomic force microscopy, interferometry or other techniques capable of determining relative depths on a sample surface. The sample can also be interrogated with other techniques such as scanning electron microscopy to characterize the corrosion behavior. These further analyses can determine if the sample corroded homogenously and possible formation of secondary phases or leached layers. Occurrence of these features may impact the accuracy of glass dissolution. This test method does not address these solid-state characterizations.

6. Procedure

6.1 *Sample Preparation:*

6.1.1 Monolithic glass samples are used in SRCA testing and require at least one flat face for surface recession measurements. The dimensions and shape of the monolith are irrelevant, although it must be able to fit though the sample ports of the reaction vessel. A coupon size of ~15 mm by 5 mm by 2 mm has been found to be convenient for the example vessel shown in Fig. 1.

6.1.2 The surface finish of the flat face(s) of the monolith is important and should be polished to ensure that the surface

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