



Designation: C1662 – 24

Standard Test Method for Measurement of the Dissolution Rate of Nuclear Waste Forms Using the Single-Pass Flow-Through Test Method¹

This standard is issued under the fixed designation C1662; 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 single-pass flow-through (SPFT) test method that can be used to measure the dissolution rate of nuclear waste forms (simulated or actual), including, but not limited to, homogenous or heterogeneous glass or glass ceramics, in various test solutions at temperatures less than 100 °C. Tests may be conducted under conditions in which the effects from dissolved species on the dissolution rate are minimized to measure the forward dissolution rate at specific values of temperature and pH, or to measure the dependence of the dissolution rate on the concentrations of various solute species.

1.2 Tests are conducted by pumping solutions in either a continuous or pulsed flow mode through a reaction cell that contains the test specimen. It is recommended that tests are conducted at several solution flow rates to evaluate the effect of the flow rate on the waste form dissolution rate.

1.3 This test method excludes static test methods in which flow is simulated by manually removing solution from the reaction cell and replacing it with fresh solution.

1.4 Tests may be conducted with demineralized water, chemical solutions (such as pH buffer solutions, simulated groundwater solutions, and brines), or actual groundwater.

1.5 Tests may be conducted with crushed waste form of a known size fraction or monolithic specimens having known geometric surface area. The reacted solids may be examined to provide additional information regarding the behavior of the waste form in the test and the reaction mechanism.

1.6 Tests may be conducted with waste forms containing radionuclides. However, this test method does not address safety issues for radioactive samples.

1.7 Data from these tests can be used to determine the values of kinetic model parameters needed to calculate the

waste form corrosion behavior in a disposal system over long periods (for example, see Practice C1174).

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

1.9 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.10 *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.*

1.11 *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

C92 Test Methods for Sieve Analysis and Water Content of Refractory Materials

C162 Terminology of Glass and Glass Products

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

C1174 Guide for Evaluation of Long-Term Behavior of Materials 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

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 www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

C1285 Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)

C1463 Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D1293 Test Methods for pH of Water

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Please refer to Terminologies **C162** and **C859** for additional terminology which may not be listed below.

3.2 Definitions:

3.2.1 *effluent solution, n*—the solution exiting the reaction cell.

3.2.2 *fines, n*—small pieces of waste form that adhere to the waste form particles prepared for use in the test that are not removed by sieving.

3.2.3 *high-purity water, n*—ASTM Type I or Type II water with a maximum total matter content including soluble silica of 0.1 g/m³ and a minimal electrical resistivity of 16.67 MΩ•cm at 25°C (see Specification **D1193** and Terminology **D1129**).

3.2.4 *influent solution, n*—the solution entering the reaction cell.

3.2.5 *leaching, n*—the preferential loss of soluble components from a waste form.

3.2.6 *mesh size fraction, n*—a designation of the size range of crushed waste form given by the combination of the smallest mesh size that the waste form is passed through (prefixed by a negative sign) and the largest mesh size that it does not pass through (prefixed by a positive sign). For example, the −40 +60 mesh size fraction will pass through a 40 mesh sieve but will not pass through a 60 mesh sieve.

3.2.7 *reaction cell, n*—the container in which the sample remains during the test.

3.2.8 *secondary phase, n*—any phase that is not present in the waste form being tested that is formed in solution or on the surface of the sample or apparatus by combination of components released from the waste form as it dissolved or present in the test solution.

3.2.9 *steady-state, adj*—in this standard, the condition in which the concentration of a dissolved waste form component remains constant due to the opposing effects of solution flow to remove the components from the vicinity of the sample and waste form dissolution to add components to solution. In the present context, dissolution of the waste form may proceed at a steady-state rate that is fixed by the solution flow rate, temperature, solution pH, and other rate-affecting processes.

3.2.10 *test solution, n*—the solution entering the reaction cell.

4. Summary of Test Method

4.1 Crushed or monolithic waste form specimens with a known surface area are contacted by a solution that continuously flows at a known flow rate and at a constant temperature through a reaction cell that contains the waste form sample. The concentration of a soluble waste form component (*i*) in the effluent solution exiting the sample cell is used to calculate the amount of waste form that has dissolved. The flow rate is determined by dividing the mass of solution that is collected for analysis by the duration over which it was collected. The dissolution rate of the waste form is calculated by using Eq 1:

$$\text{rate} = \frac{[C_i(i) - C_i^\circ] \cdot \left(\frac{F}{S^\circ}\right)}{f_i} \quad (1)$$

where $C_i(i)$ is the steady-state concentration of component *i* measured in the effluent solution, C_i° is the background concentration of component *i* in the influent solution measured in a blank test, *F* is the solution flow rate, S° is the initial surface area of the waste form sample that is exposed to solution, and f_i is the mass fraction of component *i* in the waste form. Several samples of the effluent solution are collected during the test to determine the steady-state concentrations of dissolved waste form components at a particular solution flow rate. Because the waste form dissolution rate will likely be affected by the steady-state concentrations of dissolved silica and other solutes, tests must be conducted at several solution flow rates to provide data that can be extrapolated to zero concentration to determine the forward waste form dissolution rate at infinite dilutions.

5. Significance and Use

5.1 This test method provides a prescriptive description of the design of a SPFT test apparatus and identifies aspects of the performance of SPFT tests and interpretation of test results that must be addressed by the experimenter to provide confidence in the measured dissolution rate.

5.2 The SPFT test method described in this test method can be used to characterize various aspects of waste form corrosion behavior that can be utilized in a mechanistic model, for example, calculating long-term behavior of a nuclear waste glass.

5.3 Depending on the values of test parameters that are used, the results of SPFT tests can be used to measure the intrinsic dissolution rate of a waste form, the temperature and pH dependencies of the rate, and the effects of various dissolved species on the dissolution rate.

5.4 The reacted sample recovered from a test may be examined with surface analytical techniques, such as scanning electron microscopy, to further characterize the corrosion behavior. Such examinations may provide evidence regarding whether the waste form is dissolving stoichiometrically, if particular leached layers and secondary phases were formed on the specimen surface, and so forth. These occurrences may impact the accuracy of the waste form dissolution rate that is measured using this method. This test method does not address the analysis of solid reaction materials.

6. Procedure

6.1 Fig. 1a shows a block diagram for a generic SPFT test assembly. The components of the system include a solution reservoir, transport lines, a pump, a reaction cell, and a collection bottle. The test solution is pumped from a reservoir through a reaction cell that contains the sample by a peristaltic pump or similar device. Depending on the temperature of interest, the reaction cell may be located in a constant temperature oven or water bath. The leachant must be heated to the test temperature by placing the reservoir in the same oven or by adding sufficient tubing in the oven so that the leachant achieves the test temperature before entering the reaction cell. As influent solution is pumped into the reaction cell, an equal

volume of effluent solution will be displaced from the reaction cell. The effluent solution is sampled several times during the test for analysis. The mass of effluent that is collected for analysis and the collection time are used to calculate the solution flow rate for that aliquot. Chemical analysis of the effluent solution is performed to measure the concentration of the components used to calculate the dissolution rate. The concentrations of several waste form components can be tracked to determine whether the waste form is dissolving stoichiometrically. Separate tests are conducted at several flow rates and with several sample surface areas to measure the effect of the solution composition on the measured waste form dissolution rate.

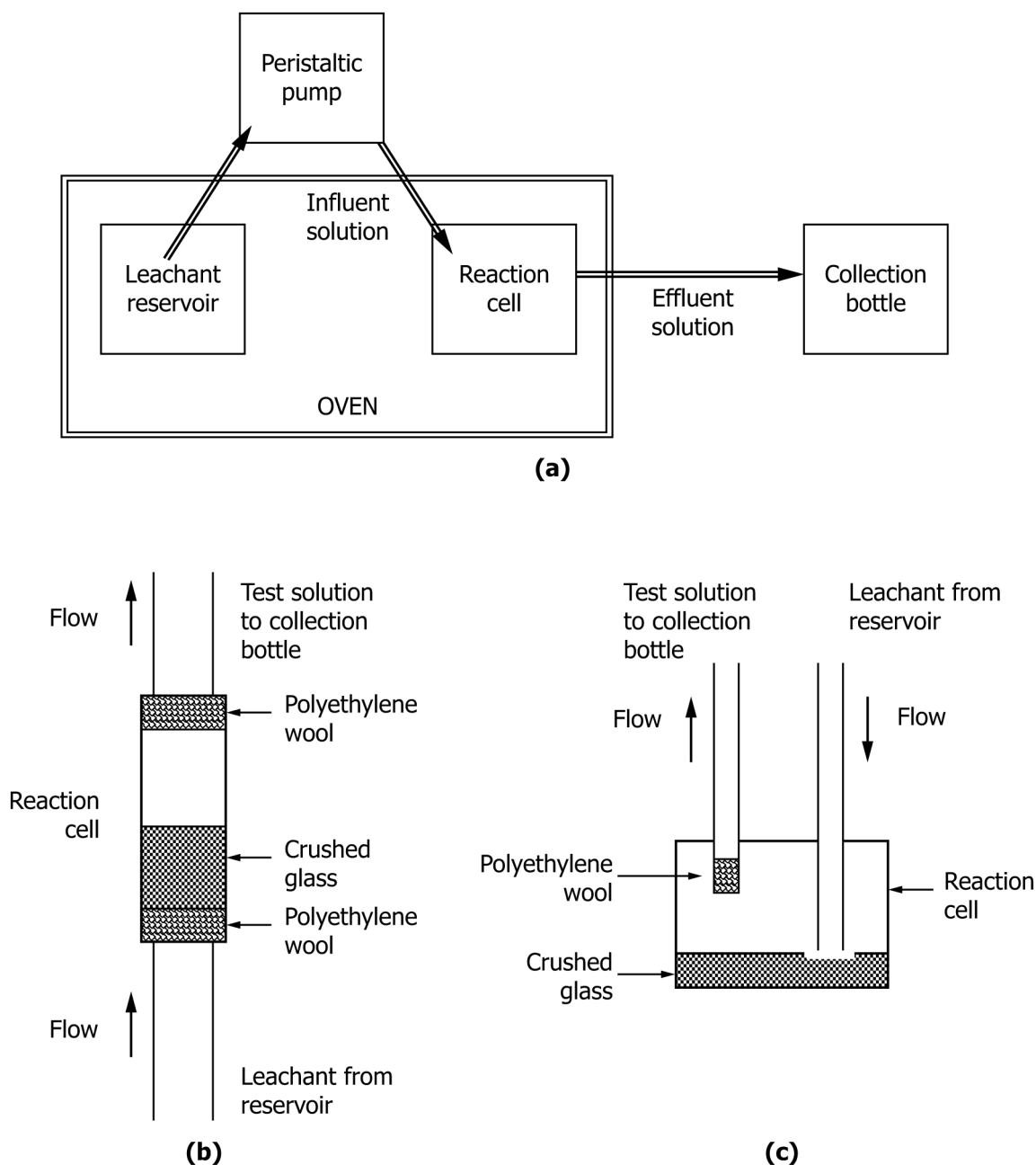


FIG. 1 (a) Schematic SPFT Design, (b) Basic Column Reactor Design and (c) Bottle Reactor Design

6.2 Either column-type (Fig. 1b) or bottle-type reaction cells (Fig. 1c) can be used; these are shown schematically in Fig. 1. In the column cell design, the influent solution is pumped (usually upwards) through the crushed waste form (or around a monolithic sample). In the bottle design, the influent solution is pumped into a cell filled with solution and displaces an equal volume of effluent solution. Polyethylene wool or an equivalent material can be used to prevent crushed waste form particles from being flushed from the reaction cell during the test, or the effluent solution can be filtered after it is collected.

6.3 Crushed waste forms can be used to provide high surface area samples. Crushed waste forms are to be prepared following the procedure for crushed sample preparation in Test Method C1285 (see Section 19 in C1285; see also Test Methods C92 and C429 for sieving methods). The surface area of crushed and sieved waste form is estimated based on the size fraction that is used in the test. The particle size of crushed samples must be large enough that the decrease in surface area during the test is less than 30 percent. The initial surface area can be calculated from the specific surface area and using the arithmetic average of the sizes of the sieve mesh and the density of the waste form (see Appendix X1 in C1285). The final surface area can be calculated based on the amount of waste form that dissolved during the test, if the particles can be modeled to have geometric shapes. The crushed waste form used in a series of SPFT tests must be from the same source to represent the homogeneity of the waste form on the scale of the test sample size. (A series of SPFT tests refers to tests conducted with the same waste form and test solution but with different masses of waste form or at different flow rates.) It is recommended that a small amount of the crushed waste form be examined with a scanning electron microscope prior to testing to document the size of the particles and the absence of fines. For porous waste forms, the surface area may need to be estimated using an analytical method capable of measuring surface area such as BET.

6.4 Monolithic samples can be used to provide samples with low surface areas. Samples can be prepared with any shape for which the geometric surface area can be measured directly. Monolithic samples are to be prepared following the sample preparation procedure in Test Method C1220 (see Section 8 in C1220). Enough monolithic glass samples shall be prepared for use in a series of SPFT tests. The surface finishes of the monoliths to be used in the series of tests shall be consistent and shall be reported with the test results. For example, if the faces of the samples are polished with silica carbide paper, the grit and lubricating fluid shall be reported. It is recommended to use a finish of 600-grit for the monolithic samples.

6.5 The mass fractions of elemental silicon in the glass must be known to determine the glass dissolution rate (see also 9.4.5). This may be determined by direct analysis of the glass (see Test Method C1463) or based on the as-batched composition of the glass. Tracers for other waste forms must be selected based on the knowledge of the phases and chemistry of the material.

6.6 The flow rate of the solution through the reaction cell is calculated by dividing the mass of test solution collected by the

duration over which it was collected. Although the flow rate is set before the sample is placed in the reaction cell, the flow rate measured with the sample in place is used for the calculations. The flow rate is likely to vary slightly with each aliquot that is taken during a test. A test is acceptable if the average test flow rate determined during a test varies by 10 % or less. Samplings in which the flow rate differs by more than 10 % shall not be used in determining the steady-state concentrations for that test.

6.7 A small change in the solution concentration may occur over time due to a change in waste form surface area. The surface area may decrease as the sample dissolves. Changes in the solution concentrations of the elements of interest with subsequent samplings (that is, with reaction time) should be noted.

6.8 It is recommended to conduct a control test under the same conditions of temperature, flow rate, and test solution that are used in the tests with the waste form, except that no sample is included in the test. The average of the solution concentrations of an element I that are measured in the control test is to be used as background concentration for element i in tests with waste forms, that is, C_i^o in Eq 1. The solution from the control test is also used to confirm that neither the influent solution nor the effluent solution was contaminated due to interactions with the apparatus.

6.9 The steady-state condition established in a flow-through test depends on the dissolution rate of the waste form and the flow rate of test solution through the reaction cell. The waste form dissolution rate will depend on the concentrations of dissolved waste form components in the solution contacting the waste form and, therefore, on the rate at which these solutes are flushed from the vicinity of the sample.

6.9.1 An upper threshold value of the flow-rate-to-surface-area exists for which a glass will dissolve at a maximum rate at a particular temperature and pH. Further increases in the flow rate will not affect the glass dissolution rate, although the steady-state concentrations will become lower, and possibly below the limit of quantification, as the flow rate is increased. The value of the threshold flow rate depends on the glass dissolution rate in the particular test conditions.

6.9.2 A lower threshold value of the flow-rate-to-surface-area exists below which the glass dissolution rate will be independent of the flow rate and will dissolve as if the system was static. Further decreases in the flow rate will not affect the glass dissolution rate. The value of the threshold flow rate depends on the glass dissolution rate in the particular test conditions.

6.9.3 The glass dissolution rate is expected to vary with the solution flow-rate-to-surface-area when the flow-rate-to-surface-area is between these extreme values.

6.9.4 As multi-phase waste forms contain phases that will presumably dissolve at different rates depending on the solution flow-rate-to-surface-area, the specifics listed in 6.9.1 – 6.9.3 may not be applicable.

6.10 To determine the forward dissolution rate, tests must be conducted at several flow-rate-to-surface-area to measure the effect of the flow-rate-to-surface-area on the dissolution rate.