



Designation: **C1308 – 08 (Reapproved 2017) C1308 – 21**

Standard Test Method for Accelerated Leach Test for Diffusive Measuring Contaminant Releases from Solidified Waste and a Computer Program to Model Diffusive, Fractional Leaching from Cylindrical Waste Forms From Solidified Waste¹

This standard is issued under the fixed designation C1308; 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 provides ~~procedures~~ a procedure for measuring the ~~leach~~ release rates of elements from a solidified matrix material, ~~determining if the material under conditions that mitigate solution feedback effects. Results can be analyzed by using different models to determine if the elemental releases are controlled by mass diffusion, computing values of diffusion constants based on models, and verifying projected long-term diffusive releases.~~ transport through the matrix (that is, by diffusion), by a surface dissolution process, or by a combination of processes. This test method is applicable to any material that does not ~~degrade or~~ deform during the test.

1.1.1 If mass diffusion is the dominant ~~step~~ process in the ~~leaching~~ release mechanism, then the results of this test can be used to calculate diffusion coefficients using mathematical diffusion models. A computer program developed for that purpose is available as a companion to this test method ~~(derive diffusion coefficients for use in diffusion-based mathematical models. Note 1)~~.

1.1.2 If surface dissolution is the dominant process, then the results of this test can be used to derive the kinetic dissolution rate in the absence of reaction affinity effects for use in dissolution-based mathematical models.

1.1.3 ~~It should be verified that leaching~~ If release is controlled by diffusion by a means other than analysis of the leach test solution data. Analysis of concentration profiles of species of interest near the surface of the solid waste form after the test is recommended for this purpose. ~~coupled or combined dissolution and mass transport processes, then the results of this test can be used to derive effective coefficient values for a mechanistic or empirical model.~~

1.1.3 ~~Potential effects of partitioning on the test results can be identified through modeling, although further testing and analyses are required to determine the cause of partitioning (for example, if it occurs during production of the material or as a result of leaching).~~

1.2 The method is a modification of other semi-dynamic tests such as the IAEA test Tests at (1) and the ANS-16.1 Leach Test wherein elevated temperatures are used to accelerate ~~diffusive~~ the release to an extent that would only be reached after very long times at lower temperatures. This approach provides a mechanistic basis for calculating diffusive releases at repository-relevant process to determine the temperature range over which the release mechanism does not change and to generate results that can be used for calculating releases at lower temperatures over long times, provided that the ~~leaching~~ release mechanism does not change with temperature.

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.

Current edition approved Jan. 1, 2017/ Feb. 1, 2021. Published January 2017/ April 2021. Originally approved in 1995. Last previous edition approved in 2008/2017 as C1308 – 08/ C1308 – 08 (2017). DOI: 10.1520/C1308-08R17-10.1520/C1308-21.

1.2.1 Tests can be conducted at elevated temperatures to accelerate diffusive release and provide a mechanistic basis for calculating diffusive releases that would occur at lower temperatures over long times. Tests conducted at high temperatures allow high temperatures can be used to determine the temperature dependence of the diffusion coefficient to be determined. They also demonstrate that the diffusion mechanism is rate-limiting through the measured extent of diffusive release. model coefficients.

1.2.2 Releases at any temperature can be projected up to the highest cumulative fractional release value that has been measured for that material (at any temperature), provided that the mechanism does not change. The mechanism is considered to remain unchanged over a range of temperatures if the diffusion model coefficients show Arrhenius behavior over that range.

Note 1—A computer program in which the test results are evaluated using three diffusion models is briefly described in Annex A1 and in the Accelerated Leach Test Method and User's Guide for the "ALT" Computer Program (2). The data are fit with model equations for diffusion from a semi-infinite solid, diffusion from a finite cylinder, and diffusion with partitioning of the species of interest to determine effective diffusion coefficients and quantify the goodness of fit. The User's Guide contains several typographical errors; these are identified in Annex A1.

1.2.3 Releases at any temperature within that range can be projected in time up to the highest cumulative fractional release value that has been measured for that material.

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

1.5 *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

C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste

D1193 Specification for Reagent Water

2.2 ANSI/ANS Standard:

ANSI 16.1 Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure⁴

<https://standards.iteh.ai/catalog/standards/sist/ab95b83b-dbbd-4853-96ae-fcd71a6dc49d/astm-c1308-21>

3. Terminology

3.1 Definitions:

3.1.1 *cumulative fraction leached*—the sum of the fractions of a species leached during all sampling intervals prior to and including the present interval divided by the amount of that species present in the test specimen before the test.

3.1.2 *diffusion coefficient (diffusivity)*—an intrinsic property of a species that relates (1) its concentration gradient to its flux in a given medium (Fick's first law), (2) its spatial rate of change in the direction of the concentration gradient to the time rate of change in its concentration in a given medium (Fick's second law), or (3) its mean square displacement to time in a given medium (the Einstein equation).

3.1.3 *dissolution*—the transfer of species from the solid test specimen into solution.

3.1.4 *effective diffusion coefficient (D_e)*—the diffusion coefficient as modified by other processes (for example, adsorption) or physical constraints (for example, tortuosity and constrictivity).

3.1.5 *finite cylinder (finite medium)*—a bounded body for which Fick's diffusion equation can be solved.

²The boldface numbers in parentheses refer to the list of references at the end of this standard.

⁴For 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.

3.1.6 *incremental fraction leached*—the amount of a species leached during a single sampling interval divided by the amount of that species in the test specimen before the test.

3.1.7 *leachant*—the initial solution with which a solid is contacted and into which the solid dissolves or is leached.

3.1.8 *leachate*—the final solution resulting from a test in which a solid is contacted by a solution and leaches or dissolves.

3.1.9 *leaching*—the preferential loss of components from a solid material into solution leaving a residual phase that is depleted in those components, but structurally unchanged.

3.1.10 *leaching interval*—the length of time during which a given volume of leachant is in contact with a specimen.

3.1.11 *leaching mechanism*—the set of processes that controls the rate of mass transport of a species out of a specimen during leaching.

3.1.12 *matrix material*—the solid material used to immobilize the waste or species of interest.

3.1.13 *partitioning*—the distribution of a contaminant between phases that may result in different release behaviors.

3.1.14 *reference leach test*—a leach test conducted with the material of interest under defined conditions, the results of which are used as a standard against which the results of other leach tests with that material are compared. In this test method, a reference leach test is one that is conducted at $20^{\circ}\text{C}\pm 20^{\circ}\text{C}$ using demineralized water.

3.1.15 *semi-dynamic leach test*—a leach test method in which the specimen is exposed to fresh leachant on a periodic schedule.

3.1.16 *semi-infinite medium*—a body having a single planar surface and extending infinitely in the directions parallel to the surface and in one direction normal to the surface.

3.1.17 *source term*—in this test method, the concentration of a species of interest in a specimen prior to leaching.

3.1.18 ~~*specimen volume*~~—*surface area*—for purposes of this test method, the ~~volume~~ geometric surface area of a monolithic specimen that is calculated from macroscopic measurements of its dimensions by assuming a simple geometric shape, such as a right circular cylinder.

3.1.19 ~~*surface area*~~—*specimen volume*—for purposes of this test method, the ~~geometric surface area~~ volume of a monolithic specimen that is calculated from macroscopic measurements of its dimensions by assuming a simple geometric shape, such as a right circular cylinder.

3.1.20 *waste form*—the waste material and any encapsulating or stabilizing matrix in which it is incorporated.

4. Summary of Test Method

4.1 This test method is a semi-dynamic leach test in which a ~~cylindrical specimen~~ specimen of known surface area and volume is immersed in a leachant of known volume that is completely replaced after specified one-day intervals. The concentration of an element of interest in the ~~recovered~~ test solution is measured ~~recovered~~ after each ~~exchange~~ interval is measured. ~~The fraction of a species of interest that is released into solution during each test interval divided by the initial amount of that species in the specimen is referred to as the incremental fraction leached (IFL).~~ The accumulated amount of the species of interest in-released during the intervals prior to and including the interval of interest is analyzed to determine if the release from thereferred to as the cumulative fraction leached (CFL-solid can be). ~~Changes in the described IFL using and a CFL mass diffusion model. The amount accumulated through a particular test duration is referred to as the values after each interval are analyzed to determine if the release from the solid can be cumulative fraction leached (CFL)-described using an analytical model.~~

4.2 Tests—The results of tests at a single temperature are adequate—common temperature may be used to compare the leaching/release behaviors of different species from the same or different materials.

4.2.1 The derived model parameter values can be used to calculate releases from waste forms with shapes different than that of the test specimen.

4.2.2 The release model optimized by regression to test results can be used to calculate releases from full-scale waste forms for long times.

4.3 The results of tests at repository-relevant temperatures can be extrapolated to long times if data from tests run at elevated temperatures and data from tests run at the reference temperature (20°C) can be modeled using a diffusion model and the diffusion coefficients show Arrhenius behavior. Elevated temperatures are used to accelerate the release of a species of interest, show that release is controlled by the same process over a temperature range, and determine the temperature dependence of the model coefficient values.

4.3.1 Elevated temperatures are used to accelerate the release of a species of interest and collect enough data to show that the release is controlled by diffusion and determine the value of the diffusion coefficient.

4.3.1 Tests must be performed at a minimum of three temperatures to verify that the leaching mechanism does not change over that temperature range.

4.3.2 By generating data/conducting tests over a range of temperatures, an Arrhenius plot can be produced to interpolate values of the diffusion coefficient/test parameters within the temperature range that was tested. Values cannot be extrapolated to temperatures that are higher or lower than the temperature range spanned by the tests.

4.3.4 A computer program that plots the experimental data and a regression curve calculated using a finite cylinder model (2) is available from ASTM (see Note 1). The program provides the value of the effective diffusion coefficient, the modeled *IFL* and *CFL* values, and a measure of the goodness of fit of the model.

4.4 If the *CFL* data from the accelerated tests, the reference test, and the fit of the modeled curve agree within defined criteria, the leaching mechanism can be taken to be diffusion-controlled and a diffusion model can be used to calculate releases from full-scale waste forms for long times. values of tests conducted at low temperatures can be extrapolated to long times if data from tests run at two or more elevated temperatures and at the reference temperature (20 °C) can all be represented by the same model and if the model coefficient values show Arrhenius behavior.

4.4.1 The results of tests at repository-relevant temperatures can be extrapolated to long times by utilizing results from tests at elevated temperatures if data from tests run at elevated temperatures and data from tests run at the reference temperature (20 °C) can be represented using the same model and the model coefficient values show Arrhenius behavior.

4.4.2 The accelerated leach test provides the maximum cumulative fractional release to which the modeled data can be extrapolated. The maximum cumulative fractional release measured represents the maximum extent of reaction for which the consistency of the mechanism has been verified for that material.

4.4.2 Because the cumulative fraction leached is a function of the specimen surface area-to-volume ratio, the results of tests with the small-scale specimens used in the ALT directly represent leaching from large-scale waste forms having the same aspect ratio.

4.4.3 The effective diffusion coefficient can be used to calculate diffusive releases from waste forms with other shapes.

4.5 The release model coefficients can be used to calculate releases from waste forms with other shapes and sizes.

4.6 If the diffusion model does not fit the data within defined criteria, no extrapolation can be made in time. The possibility of a solubility limit to the release of the species of interest shall be addressed. This can be done by evaluating the *IFL* values: constant *IFL* or specimen size. However, other models can be applied to the data to evaluate the leaching process. values may indicate either linear release kinetics or solubility limits. The results of additional tests with shorter or longer intervals can be used to discriminate between these possibilities.

~~4.5.1 A model including diffusion with partitioning of the species of interest between phases having different release behaviors is included in the computer program (2).~~

~~4.5.2 The possibility of a solubility limit to the release of the species of interest is addressed in the computer program (2).~~

~~4.7 If the data cannot be fit with a diffusion-contaminant release model within the defined criterion, then graphical comparisons of the data are recommended for added insight: For example, a plot of the cumulative fraction leached (CFL) from ALT values from a test conducted at an elevated temperature against the CFL values from ALT-a test conducted at the reference temperature can be used to verify that the accelerated data sets are consistent with the reference data and that the accelerated test appropriately accelerates tests at elevated temperatures appropriately accelerate the release, even though the release is not diffusion-limited-kinetics cannot be represented by an analytical model.~~

5. Significance and Use

~~5.1 This test method can be used to measure the release of a component from a cylindrical solidified waste form into water at the reference temperature of 20°C and at elevated temperatures that accelerate the rate and extent of leaching relative to the values measured at 20°C.~~

~~5.2 This test method can be used to:~~

~~5.2.1 Compare releases of waste components from various types of solidification agents and formulations.~~

~~5.2.2 Determine the diffusion coefficients for the release of waste components from waste forms at a specific temperature.~~

~~5.2.3 Promote greater extents of reaction than can be achieved under expected service conditions within a laboratory time frame to provide greater confidence in modeled diffusive releases.~~

~~5.2.4 Determine the temperature dependence of diffusive release.~~

~~5.1 Fitting the experimental results with a mechanistic model allows diffusive releases to be extrapolated to long times and to full-scale waste forms under the following constraints: This test method can be used to measure the release of a component from a solidified waste form into water at the reference temperature near 20 °C and at elevated temperatures that accelerate the rate and extent of leaching relative to the values measured at the reference temperature. Results of this test method can be used to quantify an intrinsic property of a material, but should not be presumed to represent releases in specific disposal environments. Tests can be conducted under conditions that represent a specific disposal environment (for example, by using a representative groundwater) to determine effective parameter values for those conditions.~~

~~5.3.1 Results of this test method address an intrinsic property of a material and should not be presumed to represent releases in specific disposal environments. Tests can be conducted under conditions that represent a specific disposal environment (for example, by using a representative groundwater) to determine an effective diffusion coefficient for those conditions.~~

~~5.3.2 Projections of releases over long times requires that the waste form matrix remain stable, which may be demonstrated by the behavior of the specimen in ALTs at elevated temperatures.~~

~~5.3.3 Extrapolations in time and scale are limited to values that correspond to the maximum CFL value obtained in an accelerated test.~~

~~5.3.4 The mechanism must be the same at all temperatures used in the extrapolation. The same model that describes the results of tests conducted at elevated temperatures must also describe the results of tests run at the reference temperature of 20°C.~~

~~5.2 This test method can be used to:~~

~~5.2.1 Compare releases of waste components from various types of solidification agents and formulations.~~

~~5.2.2 Determine the model parameter values quantifying the release of contaminants from a waste or waste form at a specific temperature.~~

5.2.3 Promote greater extents of reaction than can be achieved under expected service conditions within a laboratory time frame to provide greater confidence in modeled contaminant releases.

5.2.4 Determine the temperature dependence of contaminant release.

5.3 Fitting the experimental results with a mechanistic model allows release behaviors to be extrapolated to long times and to full-scale waste forms under the following constraints:

5.3.1 The same model must be used to represent the results of tests conducted at elevated temperatures and at the reference temperature because the mechanism must be the same.

5.3.2 Projections of releases over long times require that the waste form matrix remain stable, which may be demonstrated by the physical robustness of specimens recovered from tests conducted at elevated temperatures.

5.3.3 Extrapolations in time at any temperature within the range tested are limited to values that correspond to the maximum CFL value that was measured.

6. Apparatus

6.1 A forced-air environmental chamber or a circulating water bath capable of controlling leachant temperatures to within ± 0.1 °C of the target test temperature shall be used.

6.2 *Balance*—The balance shall be accurate to 0.1 % of the minimum test load.

7. Reagents and Materials

7.1 *Leachant*—The leachant can be selected with regard to the material being tested and the information that is desired. ~~Demineralized ASTM Type I water, synthetic or actual groundwaters, groundwater, or chemical solutions can be used. The effects of the leachant solution on the species of interest (that is, the species for which the diffusion coefficient is to be measured) and the solid must be considered. For example, the leachant should not degrade the host solid. In general, the leachant should be devoid of the species of interest to minimize solution feedback and solubility limit effects.~~ used to evaluate release in the absence of external effects, represent a particular disposal environment, or measure specific solution feed-back effects, respectively. If the leachant does contain/contains a non-negligible amount of the species/contaminant of interest, blank tests should be conducted to provide background concentrations to calculate the amounts released from the solid by using the concentrations measured in the tests. If demineralized water is used, it must meet or exceed the standards for types II or III reagent water specified in Specification during each test interval. D1193.

7.2 *Leaching Containers*—Leaching containers shall be made of a material that does not react with the leachant, leachate, or specimen. It is particularly important to select materials that are not susceptible to plate-out of species of interest from solution. ~~High density polyethylene has been found to be a and polytetrafluoroethylene (PTFE) are suitable container material/materials.~~ The top of the container shall fit tightly to minimize evaporation. The mass of the vessel must be checked before sampling to verify that evaporative losses are less than 1 % of the leachant mass ~~(or volume)~~ over every test interval. This is especially important for tests at elevated temperatures.

7.3 *Specimen Supports*—Supports for the specimens shall be made of a material that does not react with the leachant, leachate, or test specimen and is not susceptible to plate-out. High density polyethylene and PTFE have been found to be suitable support materials. The method of support should not impede leaching by contacting more than 1 % of the surface area of the specimen. Moreover, the support should not interfere with the removal and replacement of the leachate.

7.3.1 It is often convenient to suspend the waste form from the cover of the leaching container using monofilament string.

7.3.2 Alternatively, samples can be placed on perforated or mesh stands.

7.4 *Sample/Solution Containers*—~~Containers used to hold/store aliquots of leachate for storage prior to analysis should not be susceptible to plate-out of radionuclides. The container must allow for adequate preservation of the leachate and specimen.~~ not be susceptible to plate-out of radionuclides.

7.5 *Stirrers*—Stirrers ~~are~~ may be used to homogenize the leachate solution prior to removing aliquots for analysis. This is especially important for tests conducted at the reference and low temperatures. The specimen shall not be contacted during stirring.

7.6 *Filtration Equipment*—~~If filtration of~~ All visible particulates in the leachate is required, the filter medium ~~should be~~ shall be removed from the leachate either manually or by using a filter medium that is capable of removing particulates that are ~~0.45 μm~~ 0.45 μm in diameter or larger. Disposable syringe filters are ~~recommended~~. Tests recommended, but preliminary tests must be conducted to determine if the filter and the filtration apparatus ~~adsorb/retain~~ a significant amount of the species of interest. ~~interest~~ (for example, due to sorption). It may be necessary to pre-condition each filter with a sacrificial volume of the leachate solution to saturate sorption sites in the filter.

8. Specimens

8.1 Right circular cylindrical specimens shall be used with a diameter-to-height ratio between 1:1 and 1:2. This shape is used to facilitate modeling the test results. A convenient size is 2.5 cm diameter by 2.5 cm height. Smaller specimen sizes should not be used to avoid producing nonhomogeneous samples.

8.1 To the extent possible, the ~~specimens test material~~ should be prepared using the same techniques as those used to produce full-scale waste forms. For example, the curing conditions used to prepare laboratory-scale specimens should match those used for actual waste forms as closely as possible, especially the temperatures experienced by the large waste forms. The formulation, composition, processing conditions, history, and other pertinent information regarding the material being tested that could affect performance shall be recorded. Particular attention should be paid to the distributions of contaminants of interest in the material being tested so that representative specimens are used in the tests. This may include visual assessments and compositional or radiological analyses.

8.2 Accurate determination of the amount of the species of interest in the specimen at the start of the leach test shall be made and recorded. This may be based on analysis of the source material from which the specimens are prepared or analysis of the individual specimens.

8.3 ~~Specimens~~ Test specimens shall be physically, chemically, and radiologically representative of the full-scale solidified waste form. Particular attention should be paid to ensuring that the species of interest is homogeneously distributed in the material being tested. Test specimens can be cut from a larger sample or cast individually.

<https://standards.iteh.ai/catalog/standards/sist/ab95b83b-ddbd-4853-96ae-fcd7fa6dc49d/astm-c1308-21>

8.3.1 Test specimens can be cut from a larger sample or cast individually. Specimens shall be large enough to represent the homogeneity and contaminant content of the full-scale waste form and have a geometric shape for which the surface area and volume can be determined from measured dimensions.

8.3.2 Specimen sizes should be selected based on the expected leaching behavior and test conditions. Smaller specimens can be prepared for durable materials than for less-durable materials.

8.3.3 Right circular cylindrical specimens with a diameter-to-height ratio between 1-to-1 and 1-to-2 are suitable. A convenient size is 2.5 cm diameter by 2.5 cm height.

8.3.4 Cuboid and wafer specimens are also suitable and shall have a minimum edge dimension of 0.2 cm for durable materials.

8.4 Many solids prepared by casting form a skin on the outer surface during preparation that has different characteristics than the bulk material. The effect of the skin must be determined and differentiated from the bulk property. This can be done by conducting separate tests using samples with surfaces that are representative of the structure of surfaces of large waste forms, such as surfaces that are cast against container walls, and tests with samples having cut or polished surfaces that expose the bulk material to the leachant. The effect of the skin can be determined from differences in the derived diffusion coefficients for materials with and without the skin. If a specimen is prepared in a mold, any excess material should be removed from the specimen prior to weighing it. These could include casting burrs, parting lines, riser points, tags, etc. that formed during the molding process and release agents.

8.4.1 If the quantity of the species of interest in the specimen (that is, the source term) was determined before the specimen was removed from the mold, the amount of that species that remained in the mold (plus material removed as excess) shall be determined and the amount accounted to be in the specimen adjusted appropriately.

8.4.2 Many solids prepared by casting form a skin on the outer surface during preparation that has different characteristics than the bulk material. The effect of the skin must be determined and differentiated from the bulk property. This can be done by conducting separate tests using samples with surfaces that represent the surface structures of large waste forms, such as surfaces that are cast against container walls, and tests with samples having cut or polished surfaces that expose the bulk material to the leachant. The effect of the skin can be determined from differences in the derived model parameter values for materials with and without the skin.

8.5 A minimum of three replicate tests should be conducted at each temperature if results are to be used to predict long-term behavior. The dimensions of each specimen shall be measured with a calibrated device (for example, digital calipers) to the nearest 0.01 cm. The surface area and volume of the specimen are used to calculate model parameter values from the test results.

8.5.1 For right cylinder specimens, the geometric surface area and volume are calculated by modeling the specimen as a right circular cylinder. Two measurements of the diameter shall be made at both the top and bottom of the specimen and two measurements of the height made at diametrically opposite locations to take non-parallel faces and out-of-roundness into account. Measurements shall not be made at regions with visible irregularities. The surface area is calculated by using the arithmetic averages of the measured diameters and heights.

8.5.2 For cuboid and wafer test specimens, one measurement of each edge shall be made. The geometric surface area and volume are calculated by modeling each face of the specimen as a parallelepiped.

8.6 The dimensions, weight, composition, curing history, and other pertinent information that could affect performance shall be recorded for each specimen.

8.6 Accurate determination of the amount of the species of interest. The surface area and volume can be adjusted to take into account deviations in the specimen at the start of the leach test shall be made and recorded shape from an ideal right circular cylinder or parallelepiped based on additional measurements and geometric calculations.

8.7 If a specimen is prepared in a mold, any excess material should be removed from the specimen prior to weighing it. The uncertainty in the surface area and volume of the specimens should be quantified, for example, by using the propagation of errors method or, preferably, that developed by the Joint Committees for Guides in Metrology (1)³ and promulgated by NIST (2, 3); see Annex A1.

8.8.1 If the quantity of the species of interest in the specimen (that is, the source term) was determined before the specimen was removed from the mold, the amount of that species that remained in the mold (plus material removed as excess) shall be determined and the amount accounted to be in the specimen adjusted.

9. Procedure

9.1 The dimensions of each specimen shall be measured with a calibrated device (for example, digital calipers) to the nearest 0.01 cm. At least two measurements of the diameter shall be made at the top and bottom of the specimen and two measurements of the height at diametrically opposite locations. The geometric surface area and volume are calculated by modeling the specimen as a right circular cylinder and using the arithmetic averages of the measured diameters and heights.

9.1.1 The surface area and volume of the specimen are used to calculate the diffusion coefficient (see A1.3.2.1).

9.1.2 The uncertainty in the surface area and volume of the specimens contribute to the uncertainty in the diffusion constant and should be quantified, for example, by using the propagation of errors method or, preferably, that developed by the International Committee for Weights and Measured (CIPM) and promulgated by NIST (3); see Annex A2.

9.1.3 The surface area and volume used to model the results can be adjusted to take into account deviations in the specimen shape from an ideal right circular cylinder based on additional measurements and geometric calculations.

9.1 *Leachant Volume*—The leachant volume is selected based on the specimen surface area and an estimate of the leach rate. The

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>. The boldface numbers in parentheses refer to a list of references at the end of this standard.

volume must be low enough that the solution concentrations that are generated during the test can be analyzed, but high enough that solution feedback effects on leaching are negligible (that is, so that the chemical gradient between the solid and solution remains nearly constant). The solution mass can be measured and used to calculate the volume if the solution density is known. specimen surface area-to-leachant volume ratio must remain the same for all intervals in a test series to ensure that any impacts of solution feedback and solubility effects are similar during each test interval if model coefficient values are to be derived from the test results.

9.2.1 The solution volume is not used directly in the calculation of the diffusion constant, but is used to calculate the mass of the species of interest from the measured solution concentration.

9.2.2 The specimen surface area-to-solution volume must remain the same for all test intervals in an ALT to ensure that any impacts of solution feedback and solubility limitation are similar during each test interval.

9.1.1 The specimen size and solution leachant volume must be selected by compromising the benefits of using a large specimen (ease of fabrication, uniformity of specimens, ease of sampling reacted materials, etc.) with the complications of large solution volumes (handling, analytical limitations, waste disposal, etc.).

9.2.4 The effects of solution feedback and solubility limits can be identified (or mitigated) by conducting tests at different specimen surface area-to-leachant volume ratios. Solution feedback effects are expected to be more significant at higher temperatures and surface area-to-leachant volume ratios.

9.1.2 For example, to replicate the standard conditions in the Test Method C1220 static leach test, the leachant volume (in cm³) used for each interval must be 10× the surface area of the specimen (in cm²) as calculated below:

$$\frac{\text{specimen surface area (cm}^2\text{)}}{\text{leachant volume (cm}^3\text{)}} \equiv \frac{1 \text{ cm}^2}{10 \text{ cm}^3} = 0.1 \text{ cm}^{-1} \quad (1)$$

$$\frac{\text{Specimen surface area (cm}^2\text{)}}{\text{Leachant volume (cm}^3\text{)}} \equiv \frac{1 \text{ cm}^2}{10 \text{ cm}^3} = 0.1 \text{ cm}^{-1} \quad (1)$$

9.2.5.1 This ratio requires a very large volume of water for specimens of even moderate size. For example, a 2.5 × 2.5 cm cylindrical specimen having a surface area of 29.45 cm² would require 294.5 mL of solution for each of the 11 test durations. Specimens that are much larger than this and tests at lower surface area-to-leachant volume ratios will require volumes of water that need sophisticated means of wastewater handling (such as peristaltic pumps for draining the containers), since large volumes may be too unwieldy for pouring.

9.1.3 Large volumes of leachant can make analysis challenging, even for major constituents of the specimen, and present unnecessary waste disposal costs. Under these circumstances, higher specimen surface area-to-leachant volume ratio may be used. The leach rates of some waste form materials may be low enough that a specimen surface area-to-leachant volume ratio higher than 0.1. This ratio requires a large volume of water for specimens of even moderate size. For example, a 2.5 × 2.5 cm cylindrical specimen having a surface area of 29.45 cm² must be used to generate measurable solution concentrations. would require 294.5 mL of solution for each of the 11 test durations.

9.2.7 The user must verify that solution feedback effects or solubility limits do not affect the results. Solution feedback effects (or solubility limits) are considered negligible if the same value of D_e , within experimental uncertainty, is obtained for tests conducted at different specimen surface area-to-leachant volume ratios.

9.2 Leachant Volume—The leachant volume is selected based on the specimen surface area and an estimate of the release rates of the species of interest. The volume must be small enough that the solution concentrations that are generated during the test can be analyzed, but should be large enough that solution feedback effects on the release rates are negligible (that is, so that the chemical gradients between the solid and solution are the same during each test interval). The leachant mass can be measured and used to calculate the volume if the leachant density is known.

9.2.1 Large volumes of leachant that maintain highly dilute solutions can make analysis challenging, even for major constituents of the specimen, and present unnecessary waste disposal costs. Under these circumstances, a higher specimen surface area-to-leachant volume ratio may be used.

9.2.2 The leach rates of some waste form materials may be low enough that a specimen surface area-to-leachant volume ratio

higher than 0.1 cm^{-1} must be used to generate measurable solution concentrations. In contrast, the leach rates for some waste form materials may be high enough that a specimen surface area-to-leachant volume ratio lower than 0.1 cm^{-1} must be used to avoid solution saturation effects.

9.2.3 The amount of a species released may be measured directly or calculated from the measured solution concentration and volume of leachate present at the end of the test interval. The mass of the assembled vessel is measured before and after each test interval to determine the mass of water lost due to vessel leakage during the test interval and calculate the final leachate volume.

9.2.4 The effects of solution feedback and solubility limits on the release rates can be identified by conducting tests at different specimen surface area-to-leachant volume ratios or using different exchange intervals. Solution feedback effects are expected to be more significant in tests conducted at higher temperatures, higher surface area-to-leachant volume ratios, and using longer test intervals.

9.2.5 The user must verify that solution feed-back effects or solubility limits do not affect the results. Solution feedback effects (or solubility limits) are considered negligible if the same model coefficient values (for example, D_e), within experimental uncertainty, are determined for tests conducted under different test conditions (for example, specimen surface area-to-leachant volume ratios or test interval).

9.3 Temperature—For materials and formulations that have not been tested previously, leach tests shall be conducted at a minimum of three temperatures to establish that the leach release rate increases systematically with higher temperatures. One temperature must be 20°C . test must be conducted at a temperature near 20°C to be used as a reference. The other temperatures should be selected based on knowledge of the material being tested. For example, the recommended maximum temperature is 50°C for cementitious materials, for cementitious materials is 50°C , which is below the threshold of anomalous releases observed previously (34). Temperatures above 50°C can be used if it is demonstrated that the leaching mechanism does not change. Higher temperatures may be used in tests with materials known to be stable at those temperatures.

9.3.1 The reference test shall be conducted at a temperature between 19 and 25°C and maintained within 1°C of that temperature throughout the test duration. Flexibility in the reference temperature is provided in deference to the variation in ambient temperatures and its negligible effect on the test response.

9.3.2 The temperature of the controlled-temperature device shall be stabilized at or near the desired temperature prior to test initiation. The stabilized temperature establishes the target temperature for the test.

9.3.3 The controlled-temperature device must maintain a temperature within $\pm 1^\circ\text{C}$ of the desired target temperature throughout the test (except for short-term perturbations with the vessels are placed in the device or removed for sampling). The temperature shall be recorded either both before the vessel is placed in the device at the beginning of each test interval and before it is removed at the end of each test interval.

9.3.4 The time required for the device to return to the set temperature after it is opened (for example, to emplace or remove a test vessel) should be noted, even though the vessel contents may not have attained that temperature. The time required to heat the specimen to relatively high test temperatures may should be a significant small fraction of the first two test intervals (2 and 5 hours) test interval, but is expected to be consistent for all test intervals. The leachant can be preheated to the target temperature prior to the exchange to minimize this effect.

9.4 Leachant Replacement—Leachant replacements shall take place at the following time intervals: 2 hours, 5 hours, 17 hours, and 24 hours, and then daily for the next 10 days, for This test method includes daily leachant replacements over a total test duration of 11 eleven days. The times of day at which the vessel is removed from the controlled-temperature device, the specimen is removed from the leachate and placed in the fresh leachant should be noted to the nearest minute. The times at which the vessel is removed from and emplaced in the controlled-temperature device should be noted leachant, and the vessel is placed back into the controlled-temperature device should all be recorded to the nearest minute. The use of an electric clock or a watch is adequate.

9.4.1 Test vessels shall be removed from the controlled-temperature device for leachant replacement individually to minimize the temperature change of the solution prior to it being sampled.

9.4.2 The fresh leachant may be at room temperature or pre-heated to the test temperature prior to use to minimize the time required for the test to return to the test temperature. The leachant must be consistently pre-heated (or not pre-heated) prior to all replacements during the test series.

9.4.3 The mass of the assembled vessel shall be measured before the vessel is placed in the controlled-temperature device at the start of a test interval and when the vessel is removed at the end of the test interval. The difference in mass provides a measure of the loss of water due to evaporation during the test interval (see 7.2).

9.4.4 If the specimen is suspended from the top of the container, the most convenient method for changing the leachant is to lift off the cover (with the specimen still attached) and place it on a new container with vessel that contains the appropriate volume (or mass) of fresh leachant. The new leachant may be pre-heated to the test temperature (if practical). The new container can be sealed vessel shall be sealed, weighed, and placed into the temperature-controlled environment immediately. During leachant changes, the device as quickly as possible. The specimen should be exposed to air for as short a time as possible. Rinsing the sample prior to transfer is not necessary. possible during leachant changes. It is not necessary to remove the small amount of adhering solution or rinse the sample during the transfer.

9.4.5 If the specimen is on a support stand at the bottom of the test container, a portion of the leachate can be decanted into a collection container and the sample recovered removed from the test vessel with forceps and either set aside while the vessel is emptied and fresh leachant added or placed immediately into another test container vessel with leachant or pre-heated leachant (is/do not necessary to rinse the specimen). The new test container can be sealed and specimen should be exposed to air for as short a time as possible when the solution is exchanged. The new test vessel shall be sealed, weighed, and then placed into the controlled-temperature device. device as quickly as practical.

9.4.6 The mass of the assembled vessel shall be measured before the vessel is placed in the controlled-temperature device at the start of a test interval and when the vessel is removed at the end of the test interval. The difference in mass provides a measure of the loss of leachate solution due to evaporation (see If appropriate, two additional test intervals of 8 and 16 hours can be run immediately after the series of 1-day test intervals to discriminate between constant dissolution kinetics and solution saturation effects. These shall be excluded from the data set of 1-day interval tests used to derive model 7.2); parameter values.

9.4.7 If appropriate, an abbreviated replacement schedule of four 1-day test intervals can be used for material screening purposes, but those data shall not be used to derive model parameter values.

9.5 *Acid Strip—Leachate Sampling Procedure*—At least one vessel bottom shall be subjected to an acid strip at the end of a test interval to verify that the species of interest is not sorbing to the vessel. If the amount sorbed is not negligible, the vessel shall be acid-stripped after every sampling, and the amount of the species of interest recovered in the acid strip shall be added to the amount in the leachate. Immediately after opening the test vessel, stir the leachate thoroughly and remove sample aliquots quickly to minimize any artifacts that could occur during cooling (for example, precipitation). Alternatively, decant the leachate into one or more solution bottles. Several aliquots may be required at each sampling for use in the desired analyses.

9.5.1 Discard any remaining leachate solution from the vessel and rinse with demineralized water.

9.5.2 Fill vessel with an amount of demineralized water equal to or greater than the amount of leachate that was removed.

9.5.1 Add the If the specimen is suspended from the vessel lid, place the lid on the new vessel containing the appropriate amount of concentrated ultrapure nitric acid to produce a 2 volume% acid solution. fresh water and initiate the next test interval before removing aliquots of the leachate from the old vessel for analysis.

9.5.2 Cap the container and agitate, then let settle for several minutes. If the specimen is placed on a stand at the bottom of the vessel, stir the leachate and remove aliquots of the leachate for analysis before initiating the next test interval. Alternatively, decant most of the solution into one or more clean containers and remove aliquots from that container.

9.5.3 Collect a sample of the acid strip solution for analysis. The solution aliquots should be collected and preserved in ways appropriate for the analytical technique to be employed.

9.6 *Leachate Sampling*—Immediately after opening the vessel, the old leachate should be stirred thoroughly and sampled quickly to minimize any artifacts that could occur during cooling (for example, precipitation). Several aliquots may be required at each sampling for desired analyses. The solution should be inspected for the presence of particulates prior to collecting solution aliquots for analysis. If particulates are visible in the leachate, the quantity of the species of interest associated with them must be taken into account with regard to the fractions that are released and retained in the specimen during subsequent intervals. How they are treated depends on when they were generated, which can be inferred from the similarity to the material being tested. If the particles