



Designation: C1308 – 08 (Reapproved 2017)

Standard Test Method for Accelerated Leach Test for Diffusive Releases from Solidified Waste and a Computer Program to Model Diffusive, Fractional Leaching from Cylindrical Waste Forms¹

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 for measuring the leach rates of elements from a solidified matrix material, determining if the releases are controlled by mass diffusion, computing values of diffusion constants based on models, and verifying projected long-term diffusive releases. 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 in the leaching 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 ([Note 1](#)).

1.1.2 It should be verified that leaching 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.

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 ([1](#))² and the ANS 16.1 Leach Test wherein elevated temperatures are used to accelerate diffusive 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 temperatures over long times, provided that the leaching 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. Published January 2017. Originally approved in 1995. Last previous edition approved in 2008 as C1308 – 08. DOI: 10.1520/C1308-08R17.

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

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 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.

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 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](#).

1.3 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

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

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.

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

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

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

3.1.5 *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.

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

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

3.1.8 *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.

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

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

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

3.1.12 *reference leach test*—a leach test conducted under defined conditions, the results of which are used as a standard against which the results of other leach tests are compared. In this test method, a reference leach test is one that is conducted at 20°C using demineralized water.

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

3.1.14 *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.

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

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

3.1.17 *surface area*—for purposes of this test method, the 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.

3.1.18 *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 is immersed in a leachant that is completely replaced after specified intervals. The concentration of an element of interest in the recovered test solution is measured after each exchange; this is referred to as the *incremental fraction leached (IFL)*. The accumulated amount of the species of interest in the intervals prior to and including the interval of interest is analyzed to determine if the release from the solid can be described using a mass diffusion model. The amount accumulated through a particular test duration is referred to as the *cumulative fraction leached (CFL)*.

4.2 Tests at a single temperature are adequate to compare the leaching behaviors of different materials.

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.

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.

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

4.3.3 By generating data over a range of temperatures, an Arrhenius plot can be produced to interpolate values of the diffusion coefficient 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 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

4.4 If the 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.

4.4.1 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 If the diffusion model does not fit the data within defined criteria, no extrapolation can be made in time or specimen size. However, other models can be applied to the data to evaluate the leaching process.

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).

4.6 If the data cannot be fit with a diffusion 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 conducted at an elevated temperature against the *CFL* from ALT conducted at the reference temperature can be used to verify that the accelerated data are consistent with the reference data and that the accelerated test appropriately accelerates the release, even though the release is not diffusion-limited.

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.

5.3 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:

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.

6. Apparatus

6.1 A forced-air environmental chamber or a circulating water bath capable of controlling leachant temperatures to within 1°C of the target test temperature shall be used.

6.2 *Balance*—The balance shall be accurate to 0.1 % of the 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 water, synthetic or actual groundwaters, 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. If the leachant does contain a non-negligible amount of the species 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 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 suitable container material. 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.

7.3 Specimen Supports—Supports for the specimens shall be made of a material that does not react with the leachant, leachate, or specimen and is not susceptible to plate-out. 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 Containers—Containers to hold 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.

7.5 Stirrers—Stirrers are used to homogenize the leachate solution prior to removing aliquots for analysis.

7.6 Filtration Equipment—If filtration of visible particulates in the leachate is required, the filter medium should be capable of removing particulates that are 0.45 μm in diameter or larger. Disposable syringe filters are recommended. Tests must be conducted to determine if the filter and the filtration apparatus adsorb a significant amount of the species of interest. 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.

8.2 To the extent possible, the specimens 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.

8.3 Specimens shall be 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.

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.

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.

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

8.7 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.

8.8 If a specimen is prepared in a mold, any excess material should be removed from the specimen prior to weighing it.

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.

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

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.

9.2.3 The specimen size and solution 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.

9.2.5 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:

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

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.

9.2.6 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 cm⁻¹ must be used to generate measurable solution concentrations.

9.2.7 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 value of D_e , within experimental uncertainty, is obtained for tests conducted at different specimen surface area-to-leachant volume ratios.

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 rate increases systematically with higher temperatures. One temperature must be 20°C. 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, which is below the threshold of anomalous releases observed previously (3). Temperatures above 50°C can be used if it is demonstrated that the leaching mechanism does not change.

9.3.1 The controlled-temperature device must maintain a temperature within 1°C of the desired temperature throughout the test (except for short-term perturbations with the vessels are removed for sampling). The temperature shall be recorded

either before the vessel is placed in the device at the beginning of a test interval or before it is removed at the end of a test interval.

9.3.2 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 may not have attained that temperature. The time required to heat the specimen to relatively high test temperatures may be a significant fraction of the first two test intervals (2 and 5 hours).

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 a total test duration of 11 days. The times at which 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 to the nearest minute. The use of an electric clock or a watch is adequate.

9.4.1 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 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 and placed into the temperature-controlled environment immediately. During leachant changes, the specimen should be exposed to air for as short a time as possible. Rinsing the sample prior to transfer is not necessary.

9.4.2 If the specimen is at the bottom of the test container, the leachate can be decanted into a collection container and the sample recovered with forceps and placed immediately into another test container with pre-heated leachant (is not necessary to rinse the specimen). The new test container can be sealed and placed into the controlled-temperature device.

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 leachate solution due to evaporation (see 7.2).

9.5 *Acid Strip*—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.

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.

9.5.3 Add the appropriate amount of concentrated ultrapure nitric acid to produce a 2 volume% acid solution.

9.5.4 Cap the container and agitate, then let settle for several minutes.

9.5.5 Collect a sample of the acid strip solution for analysis.

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.

9.6.1 If the specimen is suspended from the vessel lid, place the lid on the vessel with fresh water and initiate the next test interval before removing aliquots of the leachate for analysis.

9.6.2 If the specimen is placed on a stand at the bottom of the vessel, stir solution and remove aliquots of the leachate for analysis before initiating the next test interval.

9.6.3 The solution aliquots should be collected and preserved in ways appropriate for the analytical technique(s) to be employed.

9.6.4 If particulates are visible in the leachate, it is necessary to account for the quantity of the species of interest associated with them.

9.6.4.1 If the particulates form by spalling from the specimen, they should be removed prior to analyzing the solution and the species of interest associated with the spalled material should be excluded from the amount released.

9.6.4.2 If the particulates formed after the species of interest were leached, two approaches can be used. One requires filtration of the leachate and subsequent analysis of both the filtrate and the particulate material on the filter. The other is to acidify the leachate to dissolve the particulates and thereby include the associated species of interest in the analyzed solution. One or both methods can be used (for example, analyze filtered and unfiltered solutions), depending on the information desired.

9.7 Analysis and Standards—Analysis of the leachate for the species of interest shall be conducted by standard methods and using appropriate calibration standards. If necessary, standards should be prepared to match the matrix elements in the samples. For radioactive specimens, a series of waste reference solutions can be prepared by diluting an aliquot of the original solution (or waste) that was used to make the specimens for comparative analysis. The analytical results for the test samples can then be compared directly to analytical results for these reference solutions to calculate fractional releases without the need for absolute standards, detector efficiencies, or decay corrections.

9.8 Standard Test—One or more ALTs with an equivalent specimen shall be conducted at 20°C for use as a standard for comparison with ALTs conducted at other temperatures and ALTs conducted with other materials. Triplicate standard tests at 20°C are required if the results will be used to project releases to long durations or larger waste forms.

9.9 Blank Test—Depending on the species of interest, a blank test with either no specimen or with a specimen that does not contain the species of interest is recommended to provide background solutions to help detect contamination that may occur during the procedure or provide background levels for leachants that contain the species of interest.

10. Calculations

10.1 Incremental Fraction Leached—The incremental fraction of species i leached (IFL) during test interval n is calculated by using Eq 2:

$$IFL = \frac{{}_i a_n}{{}_i A_0} \quad (2)$$

where:

${}_i a_n$ = the quantity of species i measured in the leachate from the n th test interval, and

${}_i A_0$ = the quantity of species i in the specimen at the beginning of the test.

In the case of radionuclide i , both terms must be corrected for radioactive decay to the beginning of the test.

10.1.1 It may be necessary to calculate the value of ${}_i a_n$ from the measured solution concentration using the leachant volume. In that case, the uncertainty in the measured concentration and the uncertainty in the leachant and leachate volumes must be taken into account. (See Annex A2.)

10.1.2 The average rate of release for any interval can be calculated by dividing IFL by the duration of that interval. The rate can then be divided by the surface area of the specimen to obtain the average fraction released per area per time. This allows comparisons of tests conducted with samples having different surface areas.

10.2 Cumulative Fraction Leached—The cumulative fraction of species i leached through the j th interval (CFL_j) is calculated by using Eq 3:

$$CFL_j = \frac{\sum_{n=1}^j {}_i a_n}{{}_i A_0} = \sum_{n=1}^j IFL_n \quad (3)$$

NOTE 2—The indices for the species and interval are excluded for convenience hereafter.

10.2.1 Plotting the CFL value for each interval against the cumulative time provides a graphical comparison of data from various tests with each other and with modeling results. An example of this type of plot is shown in Fig. 1.

10.3 Effective Diffusion Coefficient—The results of this test method can be used to determine the effective diffusion coefficient (D_e) for the release of the species of interest based on a model. A computer program has been developed at Brookhaven National Laboratory to calculate a best fit effective diffusion coefficient (D_e) based on the equations for diffusion from a semi-infinite medium or from a finite cylinder (4). The ALT computer program also evaluates the possible influence of partitioning and solubility limits on the diffusive release. That program is available from ASTM for use with this test method (2); see also (5, 6). The computer program determines the value of the effective diffusion coefficient by regressing analytical expressions for diffusion from a semi-infinite solid and from a finite cylinder to the CFL determined from the test results. The analytical expressions are provided in Annex A1. The uncertainty in the diffusion coefficient can be calculated using the formula for diffusion from a semi-infinite solid.

10.4 Agreement with Models—The CFL values calculated using values of D_e determined from the data using the diffusion

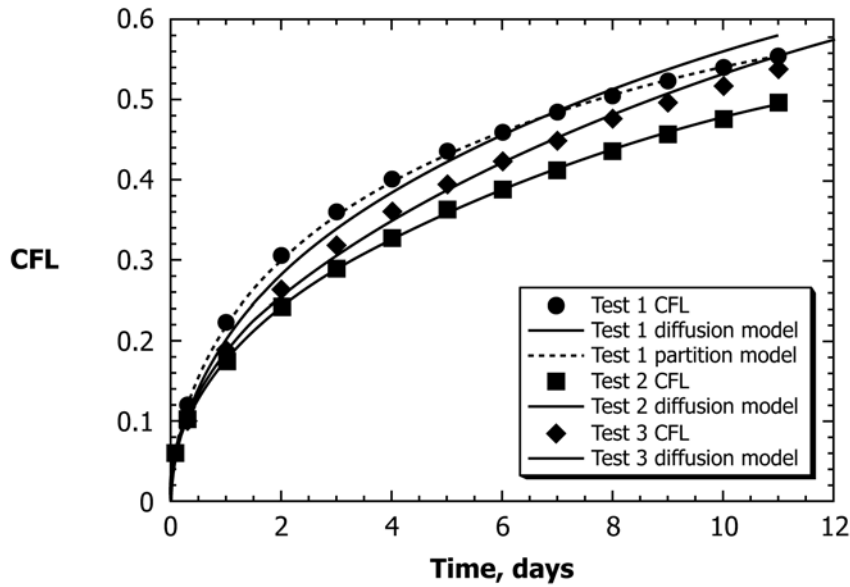


FIG. 1 Plotted Results of Test 1, Test 2, and Test 3 with Model Fits

models can be compared with the *CFL* values calculated from the test data by plotting both against the cumulative test duration. If the *CFL* values calculated with the model agree with the measured values within a designated “goodness of fit” (which is related to the uncertainty in the regression; see 10.4.1), then it can be concluded that diffusion is the rate-determining step in the leaching mechanism and the effective diffusion coefficient is the regressed value of D_e . If this is the case, then the diffusion model can be used to calculate releases over long times at that temperature. The use of the diffusion model requires that the waste form remains intact and the leaching mechanism does not change with time. Demonstrating that the same mechanism is operative at 20°C and at elevated temperatures provides confidence that it will not change over long times at intermediate temperatures, at least up to the extent represented by the maximum *CFL* value measured in a test.

10.4.1 The percent relative error in the fit of the model to the data (E_{R^2}) is determined by dividing the sum of the squares of the residuals between the *CFL* value of the optimized model curve and the measured value by the *CFL* value of the experimental data of the longest duration. For a total of N measured *CFL* values, the percent relative error for the ALT is defined as:

$$E_{R^2} = 100 \cdot \frac{\sum_{i=1}^N (CFL_{i,model} - CFL_{i,measured})^2}{CFL_{N,measured}} \quad (4)$$

10.4.1.1 A goodness of fit value of E_{R^2} equal to or less than 0.5 % is taken to mean that the diffusion model accurately represents the data. The residuals for points furthest from the mean duration are typically the highest, so the value of E_{R^2} is not conservative for the data set. Although it is not statistically unique, E_{R^2} provides a convenient empirical benchmark for the goodness of fit in tests conducted for similar total durations.

10.5 In addition to the two diffusion models, the computer program provides an indication of whether processes that complicate or mask simple diffusive release may be occurring in the ALT by using the Partition Model and Solubility Model.

10.5.1 The Partition Model divides the source term for the species of interest into separate leachable and unleachable fractions. It then uses the diffusion models to analyze release of the leachable fraction by varying the partition factor until an acceptable model fit is obtained. The Partition Model provides an effective diffusion constant, partition constant, and a measure of the relative error in the fit. An acceptable fit by the Partition Model indicates that diffusion controls the release kinetics, but that the release is complicated by an additional constraint. The species of interest may not be homogeneously distributed in the specimen or homogeneously released to solution. It may indicate an error in the surface-to-volume ratio that was used for the specimen in the calculation, or other discrepancy.

10.5.2 The Solubility Model is used to determine if solubility constraints are limiting the release of the species of interest. This could indicate that the release is not controlled by diffusion or that the testing conditions are not appropriate to measure the diffusion coefficient. The Solubility Model provides the relative standard deviation in the *IFL* values of the 1-day test intervals as the relative variance (V_R) defined as:

$$V_R = 100 \cdot \frac{\text{standard deviation}}{\text{mean IFL}} \quad (5)$$

10.5.2.1 Relative variances of 10 % or less indicate that the release is constant, within analytical uncertainties, and not diffusion-limited.

10.6 *Relationship of Temperature to Leaching*—The accelerated leach test relies on elevated temperature as the primary means of increasing the rate of mass transport from specimens.

The temperature dependence of an activated process (in this case leaching as expressed by the diffusion coefficient D_e) is usually described using the Arrhenius equation:

$$D_e = A \exp\left(\frac{k}{RT}\right) \quad (6)$$

where:

$D_e(T)$ = the effective diffusion coefficient measured at temperature T (Kelvin),

A and k = constants, and

R = the gas constant.

10.6.1 To apply Eq 6, the logarithms of the diffusion coefficients determined from experiments (D_e) conducted at several temperatures are plotted against k/T . A linear plot indicates that the increase in leaching is proportional to the increase in temperature and means that:

(1) The leaching mechanism, as well as the structural controls on leaching (for example, tortuosity, porosity), are unchanged by increasing temperature; and

(2) Effective diffusion coefficients can be calculated for temperatures between those tested.

10.7 The relationship between leaching and temperature must be determined using at least three temperatures. To project the results from short-term tests at elevated temperatures to long times at lower temperatures using this test method, it must be demonstrated that a linear relationship exists between $\log D_e$ and the inverse absolute temperature over that temperature range. The range of temperatures over which the relationship is linear defines the range for which application of the model is mechanistically justifiable. The minimum temperature is expected to be the ALT reference temperature of 20°C. The maximum temperature will likely be determined by the thermal stability of the host solid. For example, some organic matrix materials become unstable above 50°C.

10.7.1 If the value of D_e at the temperature of interest is known (by measurement or interpolation), the CFL can be calculated for long times, up to the time when the maximum CFL value measured in an ALT with that material is attained (regardless of the time or temperature at which the maximum CFL value was measured). Values of CFL projected beyond those measured in an ALT should be considered unreliable due

to possible changes in the mechanism at an extent of reaction greater than measured in a test.

10.7.2 An ALT conducted at the high temperature extreme can be continued for longer durations (additional 1-day intervals) to attain higher CFL values.

10.8 *Empirical Correlation*—If the mechanistically-based diffusion models do not provide a good fit, diffusion may not be the rate-limiting process in the leaching mechanism. Empirical approaches can be taken to compare releases from the accelerated test with releases from the reference test.

10.8.1 The effect of temperature on the release can be evaluated by plotting CFL values from the accelerated test on the y-axis of a graph and CFL values from the reference test (for the same test interval) on the x-axis. If this scatter plot shows a linear relationship, the data from the two tests can be compared and the results of the accelerated test can be said to accurately reflect the data from the reference test. The slope of the correlation provides insight regarding the effective activation energy for release. However, such empirical correlations do not confirm a diffusion-controlled mechanism and cannot be used to extrapolate the data to long times.

11. Precision and Bias

11.1 The precision of this test method will vary depending on the solid waste being tested, the temperature, and the species of interest being leached. Factors affecting the test precision include the condition of the sample surface (roughness, the presence of skin, fracturing, porosity, etc.), estimation of the geometric surface area and volume of the specimen, time at temperature, and analysis of the solutions.

11.2 No standard reference materials exist that would allow the accuracy of this test method to be determined. Determination of the precision of values discussed in this standard (expressed as the combined standard uncertainty) is discussed in Annex A2.

11.3 Results from replicate ALTs are shown in Table 1 as examples. The CFL values are plotted in Fig. 1 along with the fitted curves generated by the ALT computer model. The Diffusion Model fits for Tests 1, 2 and 3 and the Partition Model fit for Test 1 are shown. The Partition Model provides a visibly better fit than the Diffusion Model for Test 1. The sums

TABLE 1 Example ALT Test Results

Time (days)	Test 1		Test 2		Test 3	
	IFL	CFL	IFL	CFL	IFL	CFL
0.083	6.12E-02	6.12E-02	6.06E-02	6.06E-02	6.09E-02	6.09E-02
0.29	5.82E-02	1.19E-01	4.13E-02	1.02E-01	3.96E-02	1.01E-01
1.0	1.04E-01	2.23E-01	7.43E-02	1.76E-01	8.90E-02	1.90E-01
2.0	8.27E-02	3.06E-01	6.61E-02	2.42E-01	7.45E-02	2.64E-01
3.0	5.51E-02	3.61E-01	4.68E-02	2.89E-01	6.10E-02	3.25E-01
4.0	3.98E-02	4.01E-01	3.85E-02	3.28E-01	3.95E-02	3.65E-01
5.0	3.37E-02	4.35E-01	3.58E-02	3.63E-01	3.45E-02	3.99E-01
6.0	2.45E-02	4.59E-01	2.48E-02	3.88E-01	2.45E-02	4.24E-01
7.0	2.45E-02	4.84E-01	2.48E-02	4.13E-01	2.50E-02	4.49E-01
8.0	2.14E-02	5.05E-01	2.20E-02	4.35E-01	2.65E-02	4.75E-01
9.0	1.84E-02	5.23E-01	2.20E-02	4.57E-01	2.00E-02	4.95E-01
10.0	1.53E-02	5.39E-01	1.93E-02	4.76E-01	2.25E-02	5.18E-01
11.0	1.53E-02	5.54E-01	1.93E-02	4.95E-01	1.70E-02	5.35E-01

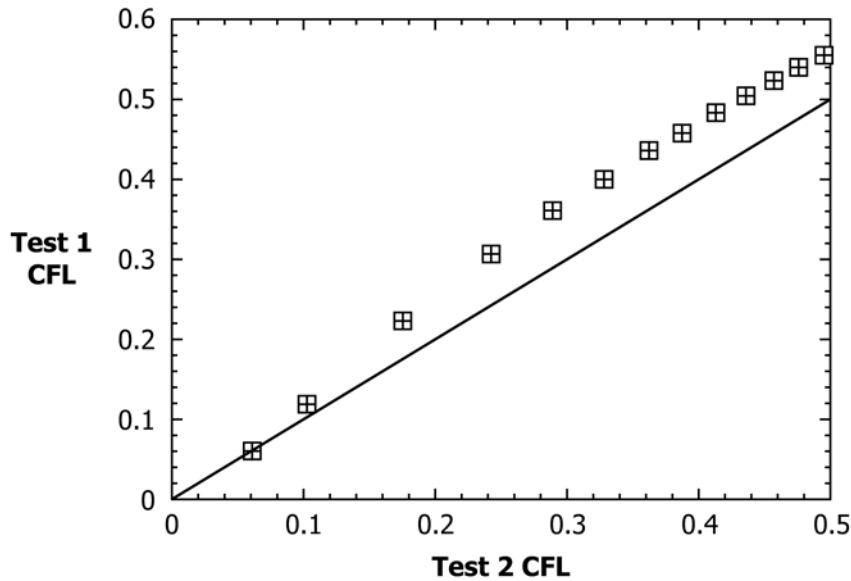


FIG. 2 Plot of the Results of Test 1 versus the Results of Test 2

of the squared residuals are 3.28×10^{-3} and 1.81×10^{-4} for the Diffusion and Partition Model fits to Test 1, respectively, and 5.62×10^{-5} for the Diffusion Model fit to Test 2. The Diffusion Model gives E_R^2 values of 0.565 % and 0.011 % for Test 1 and Test 2, respectively, and 0.06 % for Test 3. The Diffusion Model is not acceptable for the Test 1 results, based on the criterion of $E_R^2 < 0.5$, but the Partition Model gives an acceptable E_R^2 value of 0.032 for Test 1. The Diffusion Model is acceptable for the Test 2 and Test 3 results and the values of D_e are 4.98×10^{-10} m/s and 6.35×10^{-10} m/s. The improved fit for Test 1 that is obtained with the Partition Model may indicate that the value of the source term used in the Diffusion Model was too high. This could be an indication that the species of interest is not homogeneously distributed in the solid, a defect exists in the sample used in Test 1, contamination of an early sampling occurred in Test 1, etc. The value of D_e for Test 1 from the Partition Model is 2.07×10^{-9} m/s for a partition factor of 0.70. Fig. 2 shows the results of Test 1 plotted against the results of Test 2. The diagonal line in Fig. 2 shows the ideal correlation for replicate tests. In the calculated CFL value, the effect of the source term cannot be distinguished from the effect of the surface area-to-volume ratio of the test sample. By itself, the partition factor of 0.70 could indicate that the S/V ratio of the specimen used in the calculation is 43 % too low,

perhaps due to an error in the measured dimensions, the presence of micro cracks, etc. However, the observation in Fig. 2 that the differences in corresponding samplings in Test 1 and Test 2 are not linear with time or time^{1/2} suggests a real difference in the value of D_e . This suggests a difference in the surfaces of the two specimens, perhaps due to the presence of a casting film. Finally, as an example of the Solubility Model, the relative variances for samplings of Tests 1, 2 and 3 after 1 day intervals are 64.7 %, 47.4 %, and 45.0 %, respectively, which indicate that the releases in these tests are not solubility-controlled.

11.4 The results of early samplings are more heavily weighted in the determination of the diffusion coefficient than later samplings because the cumulative release fraction after each interval is used. Any error (or contamination) in a sampled concentration will be propagated to all subsequent CFL values and affect the value of D_e that is calculated.

11.5 Other data and modeling results using the ALT are available (3, 6, 7).

12. Keywords

12.1 accelerated; diffusion; leach; waste

A1. COMPUTER PROGRAM FOR THE ACCELERATED LEACH TEST

A1.1 Scope

A1.1.1 This Annex contains a brief outline of the ALT computer program that was developed to accompany the accelerated leach test. The program serves a variety of functions including:

A1.1.1.1 Comparing experimental data to curves generated by four models,

A1.1.1.2 Calculating incremental and cumulative fractional releases, and

A1.1.1.3 Storing data in a form compatible with Lotus 1-2-3.

A1.1.2 The Accelerated Leach Test computer program and a detailed Users' Guide (2) are available from: ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

A1.2 Equipment

A1.2.1 The computer program that is available for analyzing data from this test method is a compiled version and runs on IBM or IBM compatible personal computers. A math co-processor is desirable to decrease the computation time. A graphics board is required to generate plots and can be a CGA, EGA, VGA, or a Hercules color or monochrome board. In the absence of a compatible graphics board, the program will perform all calculations and list the results.

A1.3 Approach

A1.3.1 The release of components by mass transport through a solid is modeled based on the diffusion rate being proportional to the concentration gradient, as formulated in Fick's second law (Eq A1.1):

$$\frac{\partial C}{\partial t} = -D_e \nabla^2 C \quad (\text{A1.1})$$

where:

C = the concentration of the species of interest,
 t = time,
 D_e = the effective diffusion coefficient, and
 $\nabla^2 C$ = the spatial rate of change in the direction of the concentration gradient.

A1.3.2 The ALT computer program contains four mathematical models that can be used to represent the data and determine the value of the effective diffusion coefficient. The leaching mechanisms described by these models are diffusion through a semi-infinite medium, diffusion through a finite cylinder, diffusion plus partitioning of the species of interest, and solubility-limited leaching (dissolution). As illustrated in the logic flow diagram in Fig. A1.1, an iterative method is used to optimize the fit to the entire data set. The data are first fit using the semi-infinite solid medium model to obtain an initial value of D_e . If this does not give an acceptable fit, the other models are applied to the data to obtain better fits.

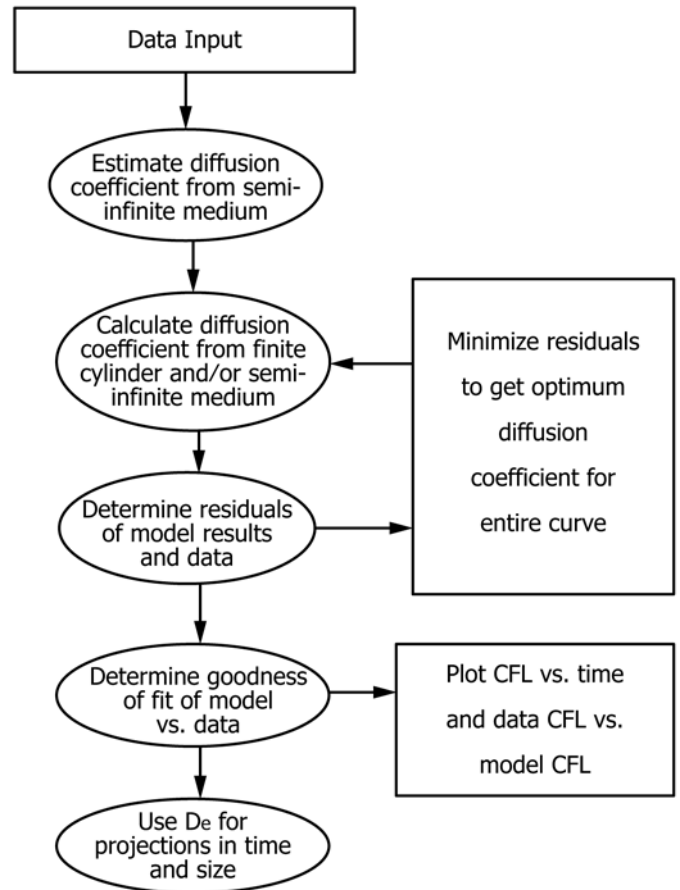


FIG. A1.1 A Flow Chart of the Major Functions of the Accelerated Leach Test Computer Program

A1.3.2.1 *Diffusion through a semi-infinite medium*—This model is usually appropriate for porous materials that give low CFL values in the ALT (for example, $CFL < 0.2$). It is the simplest model and provides an initial value of D_e for use in other models. The CFL is calculated in the semi-infinite solid model as:

$$CFL = \frac{\sum a_n}{A_0} = 2 \frac{S}{V} \left[\frac{D_e t}{\pi} \right]^{1/2} \quad (\text{A1.2})$$

where:

a_n = the total amount of the species of interest released in all leaching intervals through time t ,
 A_0 = the initial amount of the species of interest in the specimen (that is, the source term),
 S = the surface area of the specimen,
 V = the specimen volume, and
 D_e = the effective diffusion coefficient.

A1.3.2.2 *Diffusion through a finite cylinder*—This model takes into account depletion of the solid due to leaching and is usually appropriate for materials that give high CFL values in

the ALT (for example, $CFL > 0.2$). The mathematical solution is based on diffusion from a cylindrical solid of height H and radius R . In the finite cylinder model, the diffusive fractional cumulative release is calculated as a double series expression:

$$CFL = \frac{\sum a_n}{A_0} = \left(1 - \frac{32}{\pi^2} S_p(t) S_c(t) \right) \quad (A1.3)$$

with the series:

$$S_p(t) = \sum_{n=1}^{\infty} \frac{\exp\left(-\left[\frac{(2n-1)\pi}{H}\right]^2 D_e t\right)}{(2n-1)^2} \quad (A1.4)$$

and the series:

$$S_c(t) = \sum_{m=1}^{\infty} \frac{\exp\left(-\left[\frac{\beta_m}{R}\right]^2 D_e t\right)}{\beta_m^2} \quad (A1.5)$$

where the parameter β_m represents the m^{th} zero of the *zero*th order cylindrical Bessel function. Values of the β_m for $m = 1$ to 20 are provided in [Table A1.1](#). In the ALT program, an ad hoc term is added to [Eq A1.3](#) to account for the non-zero y-intercept typical in experimental results. The numerical convergence for these open series is extremely slow, and analytical closed forms expressions have been developed [\(4\)](#). The closed forms include separate terms to represent the closed series and the maximum absolute error introduced by truncating the open series. The equations developed by Pescatore [\(4\)](#) are given here for completeness.

For the $S_p(t)$ series:

$$S_p(t) = S_{p,N}(t) + E_{p,N}(t) \quad (A1.6)$$

$$S_{p,N}(t) = \sum_{n=1}^{N-1} \frac{\exp(-(2n-1)^2 \gamma^2(t))}{(2n-1)^2} + \frac{N}{(2N-1)^2} \exp[-(2N-1)^2 \gamma^2(t)] - \left(\frac{\pi^{1/2}}{2}\right) \gamma(t) \operatorname{erfc}[(2N-1) \gamma(t)] \quad (A1.7)$$

with:

$$\gamma(t) = \frac{\pi(D_e t)^{1/2}}{H} \quad (A1.8)$$

and the error term:

$$0 < E_{p,N}(t) < \frac{1}{6(2N-1)} \left[\gamma^2(t) + \frac{2}{(2N-1)^2} \right] \exp[-(2N-1)^2 \gamma^2(t)] \quad (A1.9)$$

The values n and N represent the series term and the number of terms included in the sum.

For the $S_c(t)$ series:

$$S_c(t) = S_{c,M}(t) + E_{c,M}(t) \quad (A1.10)$$

$$S_{c,M}(t) = \sum_{m=1}^{M-1} \frac{\exp(-\beta_m^2 \theta^2(t))}{\beta_m^2} \quad (A1.11)$$

$$+ \left[\frac{1}{\beta_M f_M} + \frac{1}{2\beta_M^2} \right] \exp[-\beta_M^2 \theta^2(t)] - \frac{\pi^{1/2} \theta(t)}{f_M} \operatorname{erfc}[\beta_M \theta(t)]$$

with:

$$f_M = \pi - \frac{1}{8\pi M^2}, \quad M \gg 1 \quad (A1.12)$$

$$\theta(t) = \frac{(D_e t)^{1/2}}{R} \quad (A1.13)$$

and the error term:

$$E_{c,M}(t) < \frac{f_M}{6\beta_M} \left[\theta^2(t) + \frac{1}{\beta_M^2} \right] \exp[-\beta_M^2 \theta^2(t)] \quad (A1.14)$$

The values m and M represent the series term and the number of terms included in the sum. Only the first 10 values of β_m are used in the computer program.

A1.3.3 The computer program provides plots of the experimental data and a curve calculated from the model that best fits the data. This is done through an iterative method that optimizes the fit to the entire data curve (see logic flow diagram in [Fig. A1.1](#)). Data sets are evaluated using several models sequentially and the results provided to the user.

A1.3.3.1 Diffusion Through a Semi-Infinite Cylinder—The semi-infinite solid model is used if CFL values are less than 0.2. It is also used to determine the value of the ad hoc term used to take into account the non-zero y-intercept commonly seen in diffusion tests.

A1.3.3.2 Diffusion through a Finite Cylinder—This model is used for $CFL > 0.2$. The solution to the finite cylinder equation that is used in the program was developed by Pescatore [\(4, 8\)](#). This method is particularly attractive because it becomes asymptotic at high fractional releases while using relatively little computer time. The program calculates model CFL values for the experimental test durations using various values of D_e . The optimum value of D_e is determined by minimizing the sum of the squared residuals of the measured and modeled CFL values. The relative error in fit (that is, the goodness of fit) is calculated by normalizing the sum by the squared residual of the longest-duration data point and presented as the percentage value E_{R^2} . The value of has no statistical significance, but provides a relative measure of confidence for extrapolation. In general, if the “goodness of fit” between the data curve and the model gives an E_{R^2} value less than 0.5 %, then the model can be taken to represent the leaching mechanism and can be used to calculate releases over

TABLE A1.1 Values of the Parameters β_m for $m = 1$ to 20^A

m	β_m	m	β_m	m	β_m	m	β_m
1	2.404825577	6	18.0710639679	11	33.7758202136	16	49.4826098974
2	5.5200781103	7	21.2116366299	12	36.9170983537	17	52.6240518411
3	8.6537279129	8	24.3524715308	13	40.0584257646	18	55.7655107550
4	11.7915344391	9	27.4934791320	14	43.1997917132	19	58.9069839261
5	14.9309177086	10	30.6346064684	15	46.3411883717	20	62.0484691902

^A These parameters satisfy the equation $J_0(\beta_m) = 0$, with $J_0(x)$ the zeroth order cylindrical Bessel function.

long times and scaled to calculate releases from full size cylinders. If the value of E_R is greater than 0.5 %, then the model cannot be used to make reliable projections in time or scale.

A1.3.3.3 Diffusion Plus Partitioning of the Species of Interest—This is an empirical model in which a fraction of the species of interest is not available for release to solution because of adsorption, sequestration in an alteration phase, sequestration into a more durable phase, etc. The effect of the partitioning is to decrease the amount of the species of interest in the source term (A_o) by a source term partitioning factor P , where $0 < P < 1$. The partitioned fraction may be leached at a lower rate, although this is not modeled.

$$CFL = \frac{\sum a_n}{P \cdot A_o} = 2 \frac{S}{V} \left[\frac{D_e t}{\pi} \right]^{1/2} \quad (\text{A1.15})$$

The Partition Model allows uncertainty in the concentration and distribution of the species of interest in the test material itself (that is, the source term) to be identified and taken into account. That is, the appearance of partitioning in the test results may actually indicate an error in the source term value or an artifact in the test sample. From [Eq A1.15](#), the linear impact of P on CFL can also arise due to errors in the values of S or V .

A1.3.3.4 Solubility-Limited Leaching (Dissolution)—This model accounts for systems in which diffusion is affected by the limited solubility of the species of interest, which may be established by a phase within the solid or an alteration phase formed during the test. The Solubility Model is based on the concept that the incremental fractions leached will be the same at the end of each 1-day sampling interval if the solution concentration of the species of interest is solubility-limited. Although solubility-limited release should be apparent in plots of CFL versus time and IFL versus time, the model quantifies the likelihood. The mean and standard deviation of the incremental releases are calculated and the coefficient of variation is expressed as a percentage of the mean using the following equation:

$$V_r = \frac{100}{\bar{x}} \sqrt{\frac{\sum (IFL - \bar{x})^2}{(n - 3)}} \quad (\text{A1.16})$$

where the sum is over the samplings intervals beyond the second, IFL is the incremental fraction leached during an interval, and \bar{x} is the mean of the $n-2$ IFL values that are included in the sum. Solubility is considered to limit the measured release if the coefficient of variation is less than 10 % of the mean.

A1.3.4 These processes were observed in studies with various materials during development of the test method ([3, 9](#)). Theoretical background for each mechanism is given in Appendix A of the Users' Guide ([2](#)).

A1.3.5 The results of the ALT program are presented in several forms. Tables of data and associated parameters (for example, the values of the IFL , CFL , variance, and relative error in the fit, E_R^2) are displayed on the screen and can be printed. Graphs of CFL plotted as a function of time are generated on screen and contain both experimental data points

and the curve produced by the model. In addition, graphs are available in which the experimental data are plotted on the x -axis and model-generated values are plotted on the y -axis. This type of plot allows easy comparison of the relationship between the data and the model results. If the test has been run at three or more temperatures, the activation energy (k) can be determined by the program. Projections of future releases and for full-scale waste forms can be made if diffusion is found to be the rate-limiting step in the leaching mechanism.

A1.4 Running the Program

A1.4.1 The program starts by giving the user eight options, including entering new (raw) data, entering data in the form of CFL , retrieving data from files, or editing data. Key F1 provides explanations of these choices.

A1.4.1.1 Some prompts in this program have default answers that appear in brackets. Pressing "ENTER" will select the default choice.

A1.4.2 Inputs required by various portions of the program are explained in [A1.4.2.1 – A1.4.2.10](#).

A1.4.2.1 Multiple Source Term Data—Some data require a new value for the source term for each interval. This would be necessary for a very short half-life radionuclide. The source term value that is input here is the number of counts from a standard. Corrections for dilutions are made automatically. The standard counts are separated by a comma from the leachate counts.

A1.4.2.2 Single Source Term Data—Some data require only a single value for the source term throughout the entire experiment. This can be in the form of counts per minute (CPM) or as concentration (for stable elements). For some specimens that are radioactive, liquid standards may not be available. In this case, the activity in the specimen should be calculated. This value can be input as "concentration" in the single source term option.

A1.4.2.3 Number of Sampling Increments—This is the number of samplings in the experiment. The default value is 13 for the standard ALT sampling times.

A1.4.2.4 Number of Species—This input is the number of elements or radionuclides analyzed in each set of leachate samples that need to be addressed by the program. A maximum of eight species is allowed in each data file.

A1.4.2.5 Leachate Volume—This is the volume of leachate used during each sampling interval. The test method recommends 3 litres.

A1.4.2.6 Default Times (standard ALT)—The default times are 2, 5, 17, and 24 hours for the first four intervals and the 1.0 days for the next 10 intervals, for a total test duration of 11 days.

A1.4.2.7 Sample Diameter (cm)—Diameter of the specimen that was leached.

A1.4.2.8 Sample Height (cm)—Height of the specimen that was leached.

A1.4.2.9 Counting Sample Volume (mL)—The volume of the aliquot used for radionuclide counting.

A1.4.2.10 Source Term Multiplication Factor—The factor by which the original source solution was diluted to make the counting standard. For example, 3 mL of tracer were added to

a specimen when it was made and 1 mL of that solution was diluted 1000-fold to produce the standard that was counted. The multiplication factor would be 3000 (regardless of the total volume of source solution that was made).

A1.4.2.11 Detailed instructions, in a screen-by-screen format, are given in the User's Guide (2).

A1.5 Errata in the Users' Guide (2)

NOTE A1.1—Based on comparison with reference (4), there are several typographical errors in the User's Guide for Accelerated Leach Test Computer Program (2). The following refer to equations in Appendix A of the User's Guide (2).

A1.5.1 The first term in Eq. 5 should be:

$$S_{p,N}(t) = \sum_{n=1}^{N-1} \frac{\exp(-(2n-1)^2 \gamma^2(t))}{(2n-1)^2} + \frac{N}{(2N-1)^2} \exp[-(2N-1)^2 \gamma^2(t)] - \left(\frac{\pi^{1/2}}{2}\right) \gamma(t) \operatorname{erfc}[(2N-1) \gamma(t)] \quad (\text{A1.17})$$

A1.5.1.1 There are 2 errors in the User's Guide: The first term in exponent of the first term on the right hand side $(2n-1)^2$ is incorrectly written as $(2-1)^2$ in the User Manual. The complimentary error function erfc is incorrectly written as efrc in the User Manual.

A1.5.2 The first term in Eq. 6 should be:

$$S_{c,M}(t) = \sum_{m=1}^{M-1} \frac{\exp(-\beta_m^2 \theta^2(t))}{\beta_m^2} \quad (\text{A1.18})$$

$$+ \left[\frac{1}{\beta_M f_M} + \frac{1}{2\beta_M^2} \right] \exp[-\beta_M^2 \theta^2(t)] - \frac{\pi^{1/2} \theta(t)}{f_M} \operatorname{erfc}[\beta_M \theta(t)]$$

A1.5.2.1 There are 3 errors in the User's Guide: The first term in exponent of the first term on the right hand side $\exp(-\beta_m^2 \theta^2(t))$ is incorrectly written as $\exp(-\beta_m \theta^2(t))$. Brackets around the second term

$$\left[\frac{1}{\beta_M f_M} + \frac{1}{2\beta_M^2} \right]$$

are missing. The pre-exponential in the far right term incorrectly takes the square root of $\theta(t)$.

A1.5.3 The last term in Eq. 6 (the error term) should be:

$$E_{c,M}(t) < \frac{f_M}{6\beta_M} \left[\theta^2(t) + \frac{1}{\beta_M^2} \right] \exp[-\beta_M^2 \theta^2(t)] \quad (\text{A1.19})$$

A1.5.3.1 There is 1 error in the User's Guide: The numerator of the pre-exponential term is incorrectly written as π rather than f_M .

A2. UNCERTAINTY

A2.1 It is recommended that the contributions of the parameters used to calculate a value of interest (for example, CFL , D_e , E_a) be combined to estimate the uncertainty in the calculated value following the recommendations in (8).

A2.1.1 Each component used in a calculation contributes a standard uncertainty u_i to the result. The uncertainty in a parameter may be due to a single measurement or the propagation of the uncertainties in several measurements.

A2.1.2 The parameters contributing to various values calculated within this standard are summarized in Table A2.1. The variables used to represent the parameters in this standard are included for convenience. Values of u_i are usually represented as a relative standard deviation and are assigned by the user based on experimental variables.

A2.2 The estimated standard deviation of a result y is represented by the combined standard uncertainties of the measurements that contribute to the result u_c . The combined standard uncertainty in the measurand y , $u_c(y)$, is calculated by propagating the uncertainties using the root-sum-of-squares method.

A2.2.1 For example, the surface area of a 2.5×2.5 cm right cylinder is 29.45 cm^2 . If the diameter and height are measured to a precision of 0.01 cm, then the standard uncertainty in the surface area is 0.176 cm^2 , and the relative standard deviation is 0.596 %.

A2.3 The combined standard uncertainty can be multiplied by a coverage factor, k , to represent an uncertainty range at a given confidence level. The value of k varies with the confidence level; for example, $k=2$ represents the 95.45 % confidence level. The product of k and $u_c(y)$ gives the expanded uncertainty U about the measurand y , such that any measured value Y is expected to lie within the range $y-U \leq Y \leq y+U$ at the confidence level used to calculate U .

A2.4 The combined standard uncertainty does not include systematic uncertainties such as laboratory or operator bias. It may be possible to estimate systematic uncertainties based on previous measurements in the same or other laboratory, experience with the behavior of similar materials, literature reports, and manufacturer's specifications.

TABLE A2.1 Contributions to Uncertainties in Parameters and Calculated Values

Parameter	Variable	u_i	Main Contributors to Uncertainty
Source term in specimen	A_o		Analytical uncertainty, homogeneity of source material
Specimen radius	R		Measured dimensions (half of measured diameter)
Specimen height	H		Measured dimensions
Specimen surface area	S		Measured dimensions (diameter and height); geometry
Volume of specimen	V		Measured dimensions (diameter and height); geometry
Concentration in sample	a_i		Analytical uncertainty
Volume of leachant	—		Density (if leachant volume determined by weight)
Mass of leachant	—		Measurement accuracy (if leachant volume determined by weight)
Volume or mass of counting sample	—		Measurement accuracy
Source term multiplication factor	—		Measurement accuracy, analytical uncertainty
Incremental fraction leached	IFL		Measured concentration in leachate, volume leachate, volume analytical sample, source term
Cumulative fraction leached	CFL		Uncertainty in contributing IFL
Time (interval or cumulative)	t		Precision of interval time; fraction of time at temperature
Effective diffusion coefficient (at temperature)	$D_e(T)$		CFL values, regression of CFL values, permitted deviation in regression
Temperature	T		Thermometer reading, thermocouple calibration
Activation energy	E_a		Regression of $D_e(T)$, measured T

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