



Designation: D4646 – 16 (Reapproved 2023)

Standard Test Method for 24 h Batch-Type Measurement of Contaminant Sorption by Soils and Sediments¹

This standard is issued under the fixed designation D4646; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for determining the sorption affinity of waste solutes by unconsolidated geologic material in aqueous suspension. The waste solute may be derived from a variety of sources such as wells, underdrain systems, or laboratory solutions such as those produced by waste extraction tests like the Practice D3987 shake extraction method.

1.2 This test method is applicable in screening and providing relative rankings of a large number of geomedia samples for their sorption affinity in aqueous leachate/geomedia suspensions. This test method may not simulate sorption characteristics that would occur in unperturbed geologic settings.

1.3 While this procedure may be applicable to both organic and inorganic constituents, care must be taken with respect to the stability of the particular constituents and their possible losses from solution by such processes as degradation by microbes, light, hydrolysis, or sorption to material surfaces. This test method should not be used for volatile chemical constituents (see 6.1).

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

1.5 *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.*

1.6 *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.*

¹ This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.04 on Waste Leaching Techniques.

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2. Referenced Documents

2.1 ASTM Standards:²

- D1193 Specification for Reagent Water
- D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
- D3987 Practice for Shake Extraction of Solid Waste with Water
- D4319 Test Method for Distribution Ratios by the Short-Term Batch Method (Withdrawn 2007)³
- D5681 Terminology for Waste and Waste Management
- E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
- E2551 Test Methods for Humidity Calibration (or Conformation) of Humidity Generators for Use with Thermogravimetric Analyzers

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D5681.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *distribution coefficient, K_d* —the ratio of the concentration of solute sorbed on the soil or other geomedia divided by its concentration in solution. A 24 h K_d is the analogous ratio evaluated after 24 h of contact of the solute with the geomedia.

3.2.1.1 *Discussion*—The dimensions of K_d reduce to units of volume per mass. It is convenient to express K_d in units of milliliters (or cubic centimeters) of solution per gram of geomedia. Dissimilar K_d values may be obtained if different initial solute concentrations are used, depending on the sorption behavior of the solute and the properties of the geomedia (that is, nonlinear sorption curve). This concentration dependency may be absent where the solute concentrations are sufficiently low or the characteristics of the particular solute-sorbent combination yield K_d values that are independent of the concentration of solute (that is, linear sorption curve).

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

3.2.2 *solute*—a chemical species (for example, ion, molecule, etc.) dissolved in a solution.

3.2.3 *sorbate*—a chemical species retained by a sorbent.

3.2.4 *sorbent*—a solid medium (for example, soil, sediment, till, etc.) in or upon which solutes are collected by absorption or adsorption.

3.2.5 *sorption*—a physical and chemical process by which a sorbate is taken up or held (that is, sorbed) to a sorbent by a combination of adsorption and absorption.

3.2.6 *sorption affinity*—the relative degree of sorption that occurs between a solute and a sorbent.

3.2.7 *unconsolidated geologic material (geomedia)*—a loosely aggregated solid natural material of geologic origin (for example, soil, sediment, till, etc.).

4. Summary of Test Method

4.1 Distilled water, natural water, waste leachate, or other aqueous solution containing a known concentration of a solute is mixed with a known amount of unconsolidated geologic material (geomedia) for 24 h, after which equilibrium between the sorbent and solution phase is presumed to occur. The sorbent-to-solution ratio for this test is 1:20 on a mass basis. The concentration of solute remaining in solution is measured and the amount of solute adsorbed is calculated by difference with the known concentration in the initial solute. Given that the mass of solid phase is known, the distribution coefficient, K_d , for the specified experimental conditions can then be calculated.

5. Significance and Use

5.1 This test method is meant to allow for a rapid (24 h) index of a geomedia's sorption affinity for given solutes in environmental waters or leachates. A large number of samples may be run in parallel using this test method to determine a comparative ranking of those samples, based upon the amount of solute sorbed by the geomedia, or by various geomedia or leachate constituents. The 24 h time is used to make the test convenient and also to minimize microbial, light, or hydrolytic degradation which may be a problem in longer timed procedures. While K_d values are directly applicable for screening and comparative ranking purposes, their use in predictive field applications generally requires the assumption that K_d be a fixed value.

5.2 While this test method may be useful in determining 24 h K_d values for nonvolatile organic constituents, interlaboratory testing has been carried out only for the nonvolatile inorganic species arsenic and cadmium (see Section 12). However, the procedure has been tested for single-laboratory precision with polychlorinated biphenyls (PCBs) and is believed to be useful for all stable and nonvolatile inorganic and organic constituents. This test method is not considered appropriate for volatile constituents.

5.3 The 24 h time limit may be sufficient to reach a steady-state K_d ; however, the calculated K_d value should be considered a non-equilibrium measurement unless steady-state has been determined. To report this determination as a steady-state K_d , this test method should be conducted for intermediate

times (for example, 12, 18, and 22 h) to ensure that the soluble concentrations in the solution have reached a steady state by 24 h. If a test duration of greater than 24 h is required, refer to Test Method D4319 for an alternate procedure of longer duration.

6. Interferences

6.1 When dealing with solutes of unknown stability either in contact with the geomedia or when used as blanks, care must be taken to determine if volatilization, hydrolysis, photodegradation, microbial degradation, oxidation-reduction (for example, Cr^{3+} to Cr^{6+}), or other physicochemical processes are operating at a significant rate within the time frame of the procedure. The stability and hence loss from solution may affect the outcome of this procedure if the aforementioned reactions are significant.

6.2 Although efforts should be taken to find equipment made with contact surface materials compatible with the solute solution, solute losses may occur due to sorption to material surfaces. Thus, method blanks (that is, solutes carried through the process with sorbents) should be conducted.

6.3 The compatibility of the method and the solute of interest may be assessed by determining the differences between the initial solute concentration (see 9.8) and the final method blank concentration of the solute (see 9.16). If this difference is greater than the expected precision of the method (10 %), then the K_d value generated may be unreliable and must be carefully evaluated.

7. Apparatus

7.1 *Agitation Equipment*—A device of any type that rotates about a central axis at a rate of 29 ± 2 revolutions per minute and mixes samples in an end-over-end fashion (see Practice D3987).

7.2 *Phase Separation Equipment*—A filtration apparatus made of materials compatible with the solutions being filtered and equipped with a 0.45 μm pore size membrane filter, or a constant-temperature centrifuge capable of separating particles with diameters greater than 0.1 μm (see Section 9). If organic compounds are being measured, the filtration apparatus, centrifuge tubes etc., should be compatible with the compounds being measured (for example, glass or stainless steel). Sorption of solute onto the filtration membrane may be significant for some solutes, and must be evaluated by the use of blanks through all steps of the procedure.

7.3 *Containers*—Round, wide-mouth bottles compatible with the rotary extractor and of composition suitable to the nature of the solute(s) under investigation and the analysis to be performed will be used. For nonvolatile inorganic constituents, high-density polyethylene bottles should be used with the size of the bottle dictated by sample size, and the need for the solution to occupy 70 to 80 % of the container volume (that is, 125, 250, or 2000 mL bottles for sample sizes of 5, 10, or 70 g respectively). For nonvolatile organic constituents, glass bottles or stainless steel containers with watertight closures made of chemically inert materials should be used with size requirements being the same as for nonvolatile