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## Standard Test Method for 24-h Batch-Type Measurement of Contaminant Sorption by Soils and Sediments<sup>1</sup>

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 ( $\epsilon$ ) 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 Test Method **D3987** shake extraction method.

1.2 This test method is applicable in screening and providing relative rankings of a large number of geomeedia samples for their sorption affinity in aqueous leachate/geomeedia suspensions. This test method may not exactly 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, or hydrolysis, ~~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 and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**E11** Specification for Woven Wire Test Sieve Cloth and Test Sieves

~~**D129D5681** Terminology Relating to Water for Waste and Waste Management~~

**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)<sup>3</sup>

**E2551** Test Method for Humidity Calibration (or Conformation) of Humidity Generators for Use with Thermogravimetric Analyzers

### 3. Terminology

3.1 *Definitions:*

3.1.1 For definition of terms used in this test method refer to Terminology **D129D5681**.

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

3.1.3 *sorbate*—chemical species sorbed by a sorbent.

3.1.4 *sorbent*—a substance that sorbs the solute from solution (for example, soil, sediment, till, etc.).

3.1.5 *sorption*—depletion of an amount of solute initially present in solution by a sorbent.

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

~~3.1.6 sorption affinity—the relative degree of sorption that occurs by a geomeadia.~~

~~3.1.7 unconsolidated geologic material (geomeadia)—a loosely aggregated solid natural material of geologic origin (for example, soil, sediment, till, etc.).~~

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 geomeadia 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 geomeadia.

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 ~~millilitres~~ milliliters (or cubic ~~centimetres~~ centimeters) of solution per gram of geomeadia. 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 geomeadia (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).

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 adsorption 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 (geomeadia)*—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 (geomeadia) for 24 h. ~~After 24 h, 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.~~ 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. [8497-2073058a7555/astm-d4646-16](https://standards.iteh.ai)

## 5. Significance and Use

5.1 This test method is meant to allow for a rapid (24 h) index of a geomeadia's sorption affinity for given ~~chemicals or leachate constituents.~~ solute 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 geomeadia, or by various geomeadia or leachate constituents. The 24-h time is used to make the test convenient and also to minimize ~~microbial~~ 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.~~ 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, to;~~ 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. Refer 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 geomeadia or when used as blanks, care must be taken to determine if volatilization, hydrolysis, photodegradation, microbial degradation, oxidation-reduction (for example,  $\text{Cr}^{3+}$

to  $\text{Cr}^{6+}$ ) 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. ~~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 blank concentration of the solute (see 9.15). 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.~~

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—The agitation equipment to be used is the rotary solid waste extractor.~~ A device of any type that rotates about a central axis at specified in a rate of  $29 \pm 2$  revolutions per minute and mixes samples in an end-over-end fashion (see Test Method D3987).

~~7.2 Phase Separation Equipment—A filtration apparatus made of materials compatible with the solutions being filtered and equipped with a 0.45- $\mu\text{m}$  pore size membrane filter, or a constant temperature centrifuge capable of separating particles with diameters greater than 0.1  $\mu\text{m}$  (see Section 9). If organic compounds are being measured, the filtration apparatus, centrifuge tubes etc., should be compatible with the compounds being measured (that is, 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 (Test Method D3987) 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, linear 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 mL, 250 mL, or 2-L, 125, 250, or 2000-mL bottles for sample sizes of 5, 10, or 70 g respectively). For nonvolatile organic constituents, TFE-fluorocarbon, glass bottles, or stainless steel containers with water-tight closures made of chemically inert materials should be used with size requirements being the same as for nonvolatile inorganics. Containers should be cleaned in a manner consistent with the analyses to be performed. Samples of the solutions to be analyzed should be stored in similar chemically compatible bottles.~~

~~7.4 Balance, having a minimum capacity of at least 70 g and a sensitivity of  $\pm 0.005$  g shall be used.~~

~~7.5 Mortar and Rubber-tipped Pestle—Apparatus suitable for breaking up aggregations of air-dried soil particles with breaking individual particles.~~

~~7.6 Sieve—No. 10 (2 mm) brass or stainless steel wire mesh sieve that conforms to the requirements of Specification E11.~~

~~7.7 Thermometer—A gravity thermometer conforming to the requirements of Specification E2551.~~

## 8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the American Chemical Society, where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean ~~Type IV~~ reagent water of meeting the criteria of Type IV in Specification D1193.

## 9. Procedure

9.1 Geomedia samples are spread out on a flat surface, no more than 2 to 3 cm deep, and allowed to air dry for 7 days or until constant weight (a change that is less than 5 % ~~24-h~~ over a 24-h period) is achieved. ~~(do achieved. Do not oven dry).~~ dry the geomedia.

9.2 After the sample has air dried, it is passed through a 2-mm screen sieve. Large aggregates are to be crushed, without grinding, using a clean mortar and a rubber-tipped pestle. The mass fraction of irreducible material greater than 2-mm should be noted and removed from the geomedia sample.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.