Standard Test Method for
24-Hour Batch-Type Measurement of Volatile Organic Sorption by Soils and Sediments[1]

This standard is issued under the fixed designation D 5285; 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, for example, soils, fluvial sediments, sedimentary deposits, or any other accumulations of unconsolidated solid particles (for a companion method, for metal solute, see Test Method D 4319). The waste solute may be derived from a variety of sources such as wells, underdrain systems, or laboratory solutions like those produced by waste extraction tests (for example, Test Method D 3987).

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

1.3 While this test method is intended to be applicable for all soluble organic constituents, care must be taken with respect to the stability of the particular constituents and their possible losses from solution by such processes as volatilization or degradation by microbes, light, or hydrolysis.

1.4 The values stated in SI units are to be regarded as the 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:* [2]
D 653 Terminology Relating to Soil, Rock, and Contained Fluids
D 1129 Terminology Relating to Water

D 1193 Specification for Reagent Water
D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock
D 3987 Test Method for Shake Extraction of Solid Waste with Water
D 4319 Test Method for Distribution Ratios by the Short-Term Batch Method
D 4410 Terminology of Fluvial Sediment

3. Terminology

3.1 Definitions—For definitions of terms used in this test method, refer to Terminology D 1129. For additional definitions of terms pertaining to soils and fluvial sediments, refer to Terminologies D 653 and D 4410.

3.1.1 *solute*—chemical species (for example, ion, molecules, etc.) in solution.

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

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

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

3.1.5 *unconsolidated geologic material (geomedia)*—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 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*—Dissimilar K_d values may be obtained for the same solute 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. It is absent when 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 *octanol water partition coefficient, K_{ow}*—the distribution coefficient of an organic compound between *n*-octanol and water. It has been found to be useful in predicting other

constants that describe the distribution of organics between organic carbon-containing media and water and is usually easy to measure experimentally.

3.2.3 *sorption affinity*—the relative degree of sorption that occurs by a geomedia.

3.2.4 *test compound constant, K_{oc}*— the distribution coefficient between water and a hypothetical geomedia that is 100 % organic carbon. As such, it is used to convert distribution coefficients measured for one geomedia to another geomedia with a different organic carbon content.

4. Summary of Test Method

4.1 Distilled water, natural water, waste leachate, or any aqueous solution containing a known concentration of solute is mixed with a known amount of unconsolidated geologic material (geomedia) for 24 h. After 24 h, equilibrium between the solid and solution phase is presumed to occur. The concentration of solute remaining in solution is measured. The remainder is presumed to be adsorbed onto the solid phase. Given that the mass of solid phase has previously been determined, the distribution coefficient for the specified experimental conditions can then be calculated.

5. Significance and Use

5.1 This test method is intended to allow for a rapid (24-h) index K_d of a geomedia's sorption affinity for given chemicals or leachate constituents. A large number of samples may be analyzed using this test method to determine a comparative ranking of those samples, based on the amount of solute sorbed by the geomedia, or by various geomedia or leachate constituents. The 24-h time period is used to make the test convenient as well as to minimize microbial degradation, which may be a problem in longer 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 The 24-h time limit may be sufficient to reach a steady-state K_d. However, to report this determination as a steady-state K_d, this test method should be conducted for intermediate times (for example, 12, 18, 22 h) to ensure that solute concentrations in the solution phase have reached a steady state by 24 h.

6. Interferences

6.1 When solutes of unknown stability are dealt with, when they are either in contact with the geomedia or used as blanks, care must be taken to ensure that volatilization, hydrolysis, photodegradation, microbial degradation, oxidation-reduction, or other physicochemical processes are not 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 test method and the solute of interest may be assessed by determining the differences between the initial solute concentration (see 9.3.3) and the silica sand blank concentration of the solute (see 9.3.7). If this difference is significant compared to the expected precision of the test method, the K_d value generated may be unreliable and thus must be evaluated carefully.

6.2 It is essential that the geomedia used for measuring distribution coefficients be free of any chemical species for which the distribution coefficient is to be measured. If it is suspected that the geomedia is contaminated, a procedure identical to that described in 9.3.1-9.3.7 should be followed, with Type IV water substituted for the test solution. If the concentration of a chemical in the water after equilibrating for 24 h, compared to the concentration of that chemical in the solution to be tested, is significant compared to the expected precision of the test method (± 8 %), a different geomedia should be used. Correcting the measured K_d for contaminated soil is not recommended since the preexisting contamination may not be adsorbed such that it can equilibrate readily with water.

7. Apparatus

7.1 *Agitation Equipment*—The Rotary Solid Waste Extractor[3] specified in Test Method D 3987.

7.2 *Containers*—Round, wide-mouth glass bottles that can be fitted with standard volatile organics analysis (VOA) caps and are compatible with the rotary extractor.

7.3 *Balance*, having a minimum capacity of 500 g and a sensitivity of ± 0.05 g, to be used for weighing the geomedia and solute solution. A more sensitive balance may be required for preparing analytical standards.

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 Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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 conforming to Specification D 1193.

8.3 *Silica Sand*— 20/40 grit sand blasting pure silica sand, to be used for the silica sand blank.

8.4 *Preparation of Analytical Standard Solutions*:

8.4.1 *Standard Stock Solutions*—Place approximately 9 to 9.8 mL of methanol into a 10-mL ground-glass, stoppered volumetric flask, and then allow the flask to stand unstoppered for a few minutes or until all methanol-wetted surfaces have dried. Weight the flask to the nearest 0.1 mg and immediately add a few drops of the test organic to the flask by using a 100-μL syringe, and then reweigh. Be sure that the drops fall directly into the methanol without contacting the neck of the flask. Finally, dilute to volume, stopper, and mix by inverting the flask several times.

[3] Diamondstone, B. T., Burke, R. W., and Gardner, E. L., "Improved Leach Measurements on Solid Wastes," *Standardization News*, Vol 10, No. 6, June 1982, pp. 28–33.

[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."