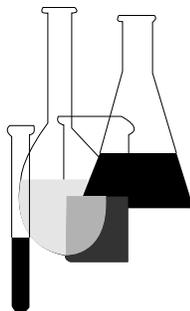




# Fate, Transport and Transformation Test Guidelines

## OPPTS 835.1220 Sediment and Soil Adsorption/Desorption Isotherm



## INTRODUCTION

This guideline is one of a series of test guidelines that have been developed by the Office of Prevention, Pesticides and Toxic Substances, United States Environmental Protection Agency for use in the testing of pesticides and toxic substances, and the development of test data that must be submitted to the Agency for review under Federal regulations.

The Office of Prevention, Pesticides and Toxic Substances (OPPTS) has developed this guideline through a process of harmonization that blended the testing guidance and requirements that existed in the Office of Pollution Prevention and Toxics (OPPT) and appeared in Title 40, Chapter I, Subchapter R of the Code of Federal Regulations (CFR), the Office of Pesticide Programs (OPP) which appeared in publications of the National Technical Information Service (NTIS) and the guidelines published by the Organization for Economic Cooperation and Development (OECD).

The purpose of harmonizing these guidelines into a single set of OPPTS guidelines is to minimize variations among the testing procedures that must be performed to meet the data requirements of the U. S. Environmental Protection Agency under the Toxic Substances Control Act (15 U.S.C. 2601) and the Federal Insecticide, Fungicide and Rodenticide Act (7 U.S.C. 136, *et seq.*).

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**OPPTS 835.1220 Sediment and soil adsorption/desorption isotherm.**

(a) **Scope**—(1) **Applicability.** This guideline is intended to meet testing requirements of both the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) (7 U.S.C. 136, *et seq.*) and the Toxic Substances Control Act (TSCA) (15 U.S.C. 2601).

(2) **Background.** The source material used in developing this harmonized OPPTS test guideline are 40 CFR 796.2750 Sediment and Soil Adsorption Isotherm and OECD 106 Adsorption/Desorption.

(b) **Guidance information**—(1) **Prerequisites.** Water solubility data; suitable analytical method; boiling point; vapor pressure curve.

(2) **Qualifying statements.** (i) This method as described is not applicable to substances which are not soluble in water to an extent which can be measured analytically. It is also not applicable to compounds which are unstable in the time scale of the test.

(ii) Although the methods can be applied to pure and commercial grade substances, the possible effects of impurities on the results should be considered.

(3) **Recommendations.** (i) It is emphasized that although three soil types are broadly described under paragraph (c)(4)(i) of this guideline, there is a wide variation of the soils used in different laboratories throughout the world and variation in the test results is to be expected. It is not possible to prescribe exactly the soils to be used, and experience with a central store of so-called “standard soil” indicates that this approach also presents problems.

(ii) This test has promised to give preliminary information on the soil leaching behavior of substances. Therefore, substances of known soil leaching behavior should be investigated. For reference substances in order to establish this possibility see paragraph (g)(4) of this guideline.

(4) **Standard documents.** The following test guideline is based on the references under paragraph (g) of this guideline. Certain modifications have been introduced to minimize the disparity in the references with regard to materials used, laboratory methodology, data presentation, and data interpretation.

(c) **Method**—(1) **Purpose, relevance, application, and limits of test.**

(i) Adsorption-desorption data are necessary for the evaluation of the migratory tendency of chemicals into the air, water, and soil or sediment compartments of our environment. They are needed to estimate, for example, leaching through the soil, volatility from water and soil, photodegradation in the adsorbed state on aerosol surfaces, and concentration in water or run-off from land surfaces into natural waters. Adsorption/

desorption processes (= sorption processes) of chemicals have an effect on transport processes and on their bioavailability. In the natural environment adsorption reduces the concentration of a chemical in aqueous solution.

(ii) Since soils have varying capacities for ion-exchange as well as differing surface areas, pH, and redox potential, interaction with chemical substances in an aqueous system is a complex process and cannot be completely defined by a simple testing scheme.

(iii) The resulting distribution between the adsorbed and solution phase depends upon chemical parameters, properties of the adsorbent and environmental parameters such as temperature, the ratio of soil to water and ionic strength. The present test method is based upon a simplified model which can serve as an initial sorption screening test for chemical substances. When warranted, more extensive testing can be performed to more accurately define the effect of a specific soil or environmental variable (for instance to obtain isotherm data)—and these possibilities are pointed out in this guideline.

(iv) The experimental procedure measures the decrease in concentration when aqueous solutions of a chemical substance are in contact with three different soil types at room temperature.

(v) A  $\text{CaCl}_2$  solution (0.01 M) is used as the aqueous solvent phase to improve centrifugation and minimize cation exchange.

**(2) Definitions and abbreviations.** (i) The definitions in section 3 of the Toxic Substances Control Act (TSCA) and the definitions in 40 CFR Part 792—Good Laboratory Practice Standards apply to this test guideline. The following definitions also apply to this test guideline.

*Soil* is the unconsolidated mixed mineral and organic material of the earth which consists of the three-phase system: Mineral and/or organic material/water/air, and which serves as a natural medium for the growth of plants.

*Sediment* is the unconsolidated material deposited at the bottom of natural water and which consists of the phases mineral material/water/(gas).

*Adsorption* in the present method does not distinguish between surface adsorption and such processes as surface catalyzed degradation, bulk adsorption, or chemical reaction.

(ii) The following abbreviations and units used in this guideline:

*a* is concentration in soil blank, in milligrams per liter (mg/L).

$c$  is decrease in concentration ( $C_i = C_e$ ) in milligrams per liter (mg/L).

$C_i$  is initial concentration (= weight of test substance/volume solution) in milligrams per liter (mg/L).

$C_e$  is final concentration, in milligrams per liter (mg/L).

$C_0$  is concentration in no-soil control, in milligrams per liter (mg/L).

$C_e$  is final concentration after correction, ( $C_e' = a$ ) in milligrams per liter (mg/L).

$d$  is volume of solution and of each wash, in liters (L).

$f$  is concentration in first desorption extraction (wash), in milligrams per liter (mg/L).

$G_A$  is quantity in no-soil control [ $C_i d(1,000)$ ] in micrograms ( $\mu\text{g}$ ).

$G_D$  is total quantity desorbed [ $(1,000)(j + 1)$ ] in micrograms ( $\mu\text{g}$ ).

$G_R$  is quantity remaining on soil after desorption ( $x - G_D$ ) in micrograms ( $\mu\text{g}$ ).

$h$  is quantity in first wash ( $g \times d$ ) in milligrams (mg).

$i$  is quantity in  $v$ , [ $C_e \times v(1/1,000)$ ], in milligrams (mg).

$j$  is quantity desorbed by first wash ( $h - i$ ) in milligrams (mg).

$k$  is concentration in second wash, in milligrams per liter (mg/L).

$l$  is quantity in second wash ( $k \times d$ ) in milligrams (mg).

$m$  is quantity of soil (oven-dried equivalent), in grams (g).

$m$  is oven dried weight of soil ( $q = pq$ ) in grams (g).

$p$  is fraction of moisture in soil.

$q$  is air dried weight of soil used, in grams (g).

$S$  is soil as described under paragraph (c)(4)(i) of this guideline.

$t$  is temperature at which test was run in degrees Celsius ( $^{\circ}\text{C}$ ).

$T$  is quantity in solution at equilibrium [ $C_e d(1,000)$ ] in micrograms ( $\mu\text{g}$ ).

$x$  is quantity adsorbed [ $(c \times d)(1,000)$ ] (or,  $G_A = T$ ) in micrograms ( $\mu\text{g}$ ).

$x/m$  is concentration adsorbed in micrograms per gram ( $\mu\text{g/g}$ ).

$V$  is volume of solution recovered from adsorption test in milliliters (mL).

$v$  is volume of solution remaining in soil ( $d - 1,000 - V$ ) in milliliters (mL).

(3) **Reference substances.** The following reference compounds need not be employed in all cases when investigating a new substance. They are listed so that the method can be calibrated from time to time and to offer the chance to compare the results when another method is applied. The values presented in the following Table 1. are not necessarily representative of the results which can be obtained with this test guideline, as they have been derived from an earlier version of it.

Table 1.—Reference Substances<sup>2</sup>

	Soil character	$K_{oc}$ <sup>1</sup>
<b>Naphthalene</b>		
Soil I .....	very strongly-strongly acid sandy soil	4205 (739–7,671)
Soil II .....	moderately or slightly acid loamy soil	7382 (772–20,088)
Soil III .....	slightly alkaline loamy soil	17,009 (1441–32,577)
<b>2,4-Dichlorophenoxy acetic acid</b>		
Soil I .....	very strongly-strongly acid sandy soil	175.9 (119.0–231.0)
Soil II .....	moderately or slightly acid loamy soil	108.4 (69.3–144.4)
Soil III .....	slightly alkaline loamy soil	225.6 (62.1 - 389.0)

<sup>1</sup> Total mean and range of mean values (in parentheses) reported by the participants of the OECD/EEC Laboratory Intercomparison Testing, Part II.

<sup>2</sup> *p*-Chloroaniline is suggested in addition to the above.

(4) **Principle of the test method.** (i) Three soils which vary significantly in cation exchange capacity, clay content, organic matter content, exchangeable cations, and pH are selected:

(A) Very strongly to strongly acid sandy soil (pH 4.5–5.5, clay content 5 percent, organic matter content 0.6–3.5 percent (e.g. Spodosol).

(B) Moderately or slightly acid loamy soil, pH 5.6–6.5; clay content 15–25 percent, organic carbon content 0.6–2.3 percent (e.g. Alfisol).

(C) Slightly alkaline loamy soil, pH 7.1–8.0;  $\text{CaCO}_3$  1–10 percent; clay content 11–25 percent, organic carbon content 0.6–2.3 percent (e.g. Entisol).

(D) All soils should be classified in terms of the US comprehensive soil classification system (on the level of the soil family) and additional information may be included (e.g. the national soil classification). The physical and chemical properties of Alfisol are given by:

## Physical and Chemical Properties of Alfisol

Soil order: .....	Alfisol
Soil series: .....	Crider
Location: .....	Gallatin County, Illinois, California
Classification: .....	typic paleudalf, fine-silty, mixed, mesic
Horizon: .....	A
percent sand: .....	1.2
percent silt: .....	86.6
percent clay: .....	12.2
percent organic carbon .....	1.0
carbonate as CaCO <sub>3</sub> .....	—
pH (1:1 H <sub>2</sub> O): .....	7.2
cation exchange capacity (meq/100 g).	13.5
extractable cations (meq/100 g).	
Ca .....	8.4
Mg .....	2.8
Na .....	—
K .....	0.7
H .....	1.6
Clay Fraction Mineralogy: .....	>50% Montmorillonite; 5–20% mica; <5% kaolinite (36–120 cm depth)

(ii) All three soils are common in temperate zones, but are not representative of arid or tropical regions. They may also be used for further testing designed to study behavior in sedimentary systems.

(iii) The method first establishes that a suitable analytical procedure exists, then proceeds to answer the question whether the chemical substance has a tendency to be strongly, weakly or nonadsorbed from a dilute aqueous solution by any of the three soil types. If little or no adsorption occurs, no further sorption tests are performed because the chemical will not be retained in soil media.

(iv) If adsorption is moderate or high ( $A > 25\%$ ), a single desorption test is performed to establish whether or not this chemical is likely to be retained in soil media. If it is readily desorbed ( $D > 75\%$ ), no further sorption tests are required; if only poorly desorbed ( $D < 75\%$ ), the screening test is considered to be complete and the investigator has the option of proceeding to the secondary or advanced phase in which an adsorption isotherm is determined and a mass balance established.

(v) Under the advanced test, the absorption kinetics are determined. If neither a plateau value nor a mass balance value is obtained, check for biotic and abiotic degradation. If a plateau value or a mass balance value is obtained, the absorption isotherms are determined. If the soil envi-

ronment is not considered a likely as a trap for the substance, no further tests are needed because the soil media are of minimal importance. The information should be considered for hazard assessment of the substance. If the soil environment seems to be a likely trap, consider the fate of the substance in soil, and consider the information for hazard assessment of the substance.

(5) **Quality criteria**—(i) **Repeatability.** The repeatability of the measurement is not well known at present, as is indicated with two reference compounds named under paragraph (c)(3) of this guideline. Standard deviation of the mean should be determined.

(ii) **Sensitivity.** The sensitivity of the test method depends on the water solubility of the test substance.

(iii) **Specificity.** This method as described is not applicable to water insoluble gases, hydrolytically unstable compounds, chemicals which may suffer catalytic decomposition on soil surface, or in cases where analytical methods are unavailable in the 0.01 mg/L range.

(iv) **Possibility of standardization.** The method itself can be standardized, but the soils probably cannot. There is no possibility of automation.

(d) **Description of the test procedure**—(1) **Preparations.** The adsorption test should be run using sieved soil to be less than or equal to 2 mm (N° 10 mesh) soil which has either been stored damp since collection or air-dried and reequilibrated with approximately twice its weight of water before use. (This reequilibration should be carried out for a minimum of 24 h by gently tumbling the soil and water together.) In either case the dry weight content (100 °C for approximately 12 h) of the soil should be determined.

(2) **Test conditions.** (i) The three soil samples selected for this study should have a pH between 4 and 8, an organic carbon content between 0.6 and 3.5 percent. Suggested soil types are described under paragraph (c)(4)(i) of this guideline.

(ii) Distilled or deionized water are used to prepare the CaCl<sub>2</sub> solution (0.01 M) as the solvent.

(iii) Containers should be made of materials that adsorb negligible amounts of the chemical to be tested. Other details of container design are not critical, although handling and transfer errors are minimized by using centrifuge tubes. The test solutions listed under paragraph (d)(3)(ii) through (iv) of this guideline should be added to their respective test containers and equilibrated (without soil sample). The containers should be closed during agitation and centrifugation to avoid volatility losses.

(iv) The actual determination should be carried out in duplicate using, e.g. 1:5 w/w soil/solution ratio.

(v) The experiment should be run at room temperature.

(3) **Performance of the tests**—(i) **Preliminary test.** (A) To assure applicability of the method, especially the analytical method, to a given chemical substance the following test should be performed.

(B) The soil is equilibrated with the aqueous phase (0.01 M CaCl<sub>2</sub>) as described under paragraph (d)(3)(ii)(A) of this guideline. The aqueous phase is separated by centrifugation, and sufficient test chemical is added to form a solution of approximately one-half saturation and not exceeding 5 mg/L, unless the analytical method cannot be operated at this level. The solution is then subjected to chemical analysis and, if satisfactory, the screening test can be performed. A satisfactory, reproducible analytical method is essential to this method.

(ii) **Screening test: Adsorption.** (A) The solution of test chemical is prepared in 0.01 m CaCl<sub>2</sub> at a level of one-half saturation but less than 5 mg/L maximum, unless otherwise dictated by the analytical method.

(B) The adsorption step is performed in duplicate upon this single concentration solution using the three soil types.

(C) Blanks include each of the three soils with only 0.01 M CaCl<sub>2</sub> solution (no test chemical) and a single control of the test chemical solution with no soil. These blanks permit correction for analytical interference due to soil extracts as well as correction for interaction between chemical and container or handling losses.

(D) The soil is added to the test solution and agitation is begun immediately. Agitation must be continuous and sufficient to maintain the adsorbent (soil) in suspension, but should not be so vigorous as to cause the breakdown of the soil structure.

(E) Agitation is continued for 16 h.

(F) After agitation, this suspension is centrifuged to obtain a clear solution. The volume of clear aqueous solution is measured and either analyzed immediately for parent compound concentration or stored under refrigeration (5 °C) to minimize biodegradation.

(G) The three blanks and control are subjected to precisely the same steps as the test systems and appropriate correction is applied as necessary.

(H) The volume of equilibrium test solution retained in the solid phase following centrifugation and the volume, V, of the supernatant must be recorded for subsequent correction of the desorption data.

(iii) **Screening test: Desorption.** (A) If no significant adsorption (approximately 25 percent or less) occurs, the desorption part of the test is not called for.

(B) If medium to high adsorption occurs ( $A > 25\%$ ), desorption testing is carried out.

(1) To each solid phase is added a volume  $V$  (see paragraph (d)(3)(ii)(H) of this guideline) of 0.01 M  $\text{CaCl}_2$  solution and this mixture gently agitated for 16 h. It is then centrifuged and the supernatant retained for analysis. The whole desorption procedure is then repeated on the solid phase with a further quantity of fresh 0.01 M  $\text{CaCl}_2$  solution.

(2) The solute retained in the entrained equilibrium test solution under paragraph (d)(3)(ii)(H) is assumed to be completely removed in the two washings along with the desorbed material and an appropriate correction applied to decrease the measured concentration by the calculated amount of retained solute.

(3) The determination of the amount and the characterization of the chemical remaining in the aqueous solution phase should be accomplished by appropriate analytical methods.

(iv) **Advanced test: Adsorption.** (A) If the screening test demonstrates a significant degree of chemical adsorptivity, adsorption isotherms may be helpful in predicting mobility in soil/water systems.

(B) To ascertain that equilibrium conditions will be met in the advanced test, a series of samples of each soil is prepared using one convenient concentration of test chemical. These samples are analyzed after appropriate time intervals. If no decomposition of the test chemical occurs, its concentration in the aqueous phase will eventually reach a plateau value. The time required to reach this plateau value is the minimum agitation time. If no plateau value can be reached abiotic or biotic degradation must be considered.

(C) For the adsorption isotherm determination, four initial concentrations,  $C_i$ , of approximately 0.04, 0.20, 1.00, and 5.00 mg/L are prepared and the adsorption test performed on each as described under paragraph (d)(3)(ii) of this guideline. In some cases the low solubility of the test chemical will prohibit the use of some of the above concentrations. In these cases the adsorption isotherm is measured using high and low (other than 0.00) equilibrium solution concentrations of the chemical that are at least 1 order of magnitude apart.

(D) Blanks are identical with those described under paragraph (d)(3)(ii)(C) of this guideline.

(E) An analytical control sample (no soil) should also be run at a convenient test concentration, not to exceed approximately 1 mg/L.

(F) Analysis and correction are as before.

(v) **Mass balance.** (A) A mass balance should be performed by determining, in addition to the concentrations in the solutions, the concentration of the substance in the soil. This additional measurement needs only be performed after reaching the equilibrium plateau and only for one concentration with each soil.

(B) The adsorbed chemical substances remaining on the soil particles are removed so that a mass balance can be attempted. The extraction medium should be a suitable solvent in which the chemical of interest is very soluble (minimum 1 mg/L). A volume of suitable solvent equal to the volume of aqueous solution originally used to attain equilibrium is added to the adsorbent and shaken vigorously for 10 min.

(C) The mixture is centrifuged to obtain a clear solution.

(D) The extraction procedure is performed 3 times with equal volumes of fresh solvent which are then analyzed.

(E) The aqueous solution phase is analyzed for the parent chemical under test.

(e) **Data and reporting**—(1) **Treatment of results**—(i) **Soil characterization.** Report data on soil using the format provided under paragraph (f) of this guideline, DATA SHEET FOR SEDIMENT AND SOIL ADSORPTION/DESORPTION ISOTHERM, part A. Soils used as adsorbents.

(ii) **Screening test.** (A) The data required are:

(1)  $m$  = dry weight of soil employed (g)

(2)  $C_e$  = concentration of substance remaining in solution (V) in the adsorption step (g/L)

(3)  $C_1$  = concentration of substance in solution in the first wash (g/L)

(4)  $C_2$  = concentration of substance in solution in the second wash (g/L)

(5)  $V_0$  = original volume of solution employed (mL)

(6)  $V$  = volume of solution obtained after the adsorption step (mL)

(7)  $G$  = quantity of material recovered from the soilless control (g)

(B) The calculations to be carried out are as follows:

(1) The percent of the chemical adsorbed is calculated as:

$$A = (G - C_e \times V_0) / G \times 100 = x / G \times 100$$

(2) The percent of the adsorbed material which is desorbed is calculated as

$$D = [(C_1 + C_2)V - (V_0 - V)C_e]/x \times 100$$

(3) The percent of the adsorbed material which is not desorbed is calculated as:

$$R = [G - (C_e + C_1 + C_2)V]/x \times 100$$

(4) The adsorption coefficient for the determination is calculated as:

$$K' = (x/m)/C_e$$

(5) The adsorption coefficient should also be calculated as a function of the organic carbon content of the soil:

$$K'_{oc} = K'(100/\text{percent organic carbon})$$

(iii) **Advanced test.** (A) The time required to reach the equilibrium concentration,  $C_e$ , should be determined.

(B) In addition to using the data to perform the calculations in the screening test under paragraph (e)(1)(ii) of this guideline, the data should be plotted according to the Freundlich equation. While the equation is empirical it is widely used and has been found to describe adequately the adsorption process in dilute solution. The equation has the form:

$$(x/m) = (K) + (C_e^{1/n})$$

$K$  and  $1/n$  are empirical constants characteristics of the test compound and the sorbent used in the test. Graphically,  $K$  is the  $x/m$  intercept of the isotherm plot at  $C_e = 1$  and  $1/n$  is the slope of the line when  $\log x/m$  is plotted on the ordinate (vertical axis) and  $\log C_e$  is plotted on the abscissa (horizontal axis). The intercept is an indicator of sorption capacity and the slope of sorption intensity.

(iv) **Mass balance.** A mass balance should be performed for the parent compound. The combined concentrations of the parent chemical in the aqueous phase and the organic solvent extracts are subtracted from the initial concentrations of parent compound and reported. A reasonable hypothesis should be submitted to account for any discrepancy significantly greater than the experimental error.

(v) **Adsorption isotherm and mass balance data format.** (A) The following format is used for presenting adsorption isotherm data:

Adsorption Isotherm Data

x/m	$C_e$	$\log (x/m)$	$\log C_e$
.....	.....	.....	.....

Adsorption Isotherm Data—Continued

x/m	C <sub>e</sub>	log (x/m)	log C <sub>e</sub>
.....	.....	.....	.....
.....	.....	.....	.....

(B) Format for graphing adsorption isotherm data for sediment or soil samples:

(1) Graph 1.—Abscissa is C<sub>e</sub> (μg in solution); ordinate is x/m (μg adsorbed per gram of soil).

(2) Graph 2.—Abscissa is log C<sub>e</sub>; ordinate is log (x/m). (Log (x/m) = log K + 1/n log C<sub>e</sub>; intercept = log K; slope = 1/n; R<sup>2</sup> = coefficient of determination for the regression analysis.

(C) The following format should be used for presentation of mass balance data.

Mass Balance Data

Data	Soil I	Soil II	Soil III
Original amount (μg) .....	.....	.....	.....
Amount absorbed at plateau (μg) .....	.....	.....	.....
Amount in equilibrium solution (μg) .....	.....	.....	.....
Unaccounted material [μg/(a - (b - c))] .....	.....	.....	.....
Percentage of unaccounted material [(b + c)/a × 100 = %] .....	.....	.....	.....

(2) **Test report.** Information should be submitted for the soils selected using the data sheet provided under paragraph (f) of this guideline.

(i) **Screening test.** Report the percent of chemical adsorbed, A, and if necessary, the percent of adsorbed material which is desorbed, D, and the percent of the adsorbed material which is not desorbed, R, for each soil. Also report K' and K'<sub>oc</sub> and the concentration C<sub>e</sub> used in the calculation (refer to paragraph (e)(1)(ii)(B) of this guideline).

(ii) **Adsorption/desorption test report** The following Tables 2. and 3. list the data required.

**Table 2.—Adsorption Test**

Data	Soil I	Soil II	Soil III
concentration of test substance (mg/L) .....	.....	.....	.....
after contact of . . . hours with soil .....	.....	.....	.....
correction for blank with soil .....	.....	.....	.....
correction for blank without soil .....	.....	.....	.....
final corrected concentration (mg/L) .....	.....	.....	.....

**Table 2.—Adsorption Test—Continued**

Data	Soil I	Soil II	Soil III
initial concentration of test solution (mg/L) .....	.....	.....	.....
decrease in concentration (mg/L) .....	.....	.....	.....
quantity adsorbed (µg) .....	.....	.....	.....
quantity of soil (g of oven-dried equivalent) .....	.....	.....	.....
quantity adsorbed (µg) per gram of soil .....	.....	.....	.....
temperature of test (°C) .....	.....	.....	.....
volume of solution recovered after centrifugation (mL) .....	.....	.....	.....
volume of solution not recovered (mL) .....	.....	.....	.....
corresponding quantity of test substance (mg) .....	.....	.....	.....

**Table 3.—Desorption Test**

Data	Soil I	Soil II	Soil III
concentration in combined washings (mg/L) .....	.....	.....	.....
corresponding quantity of test substance (mg) .....	.....	.....	.....
quantity desorbed (µg) .....	.....	.....	.....

(iii) **Advanced test.** (A) The time for reaching the plateau value should be recorded together with the raw data.

(B) The amount of adsorbed chemical (x) per unit amount of adsorbent (m) (usually expressed as micrograms per gram (µg/g) soil) is defined as x/m. This value is calculated from the difference between the initial concentration of the chemical in the solution and the equilibrium solution concentration of the chemical (C<sub>e</sub>), the volume of the solution and the weight of the soil (see paragraph (e)(2)(ii) of this guideline).

(C) Average values of the adsorbed chemical, x/m, are plotted graphically as a function of equilibrium concentration, C<sub>e</sub>, for each soil.

(D) The results can also be evaluated by plotting log (x/m) versus log C<sub>e</sub>. If the relationship is linear then the Freundlich isotherm

$$x/m = K \times C_e^{1/n}$$

may be applied in its logarithmic form

$$\log x/m = \log K + 1/n \log C_e$$

and the linear regression calculated. If the regression is significant (for N = 5, P = 5%, R<sup>2</sup> ≥ 0.77), the adsorption constant K and the constant 1/n should be reported along with all raw data (see Mass Balance under paragraph (e)(1)(iv) of this guideline).

(E) K<sub>oc</sub> should be determined from K and recorded.

(f) **Data report forms.** Sample data sheets for reporting test results are attached, and may be duplicated.

**DATA SHEET FOR SEDIMENT AND SOIL ADSORPTION/  
DESORPTION ISOTHERM**

**Laboratory:** \_\_\_\_\_

**Date:** \_\_\_\_\_

**Test Substance:** \_\_\_\_\_

Formula: \_\_\_\_\_

Name (IUPAC): \_\_\_\_\_

**A. Soils used as adsorbents**

	I	II	III
Soil order .....	.....	.....	.....
Soil series .....	.....	.....	.....
Classification .....	.....	.....	.....
Location .....	.....	.....	.....
Horizon .....	.....	.....	.....
percent sand .....	.....	.....	.....
percent silt .....	.....	.....	.....
percent clay .....	.....	.....	.....
percent organic matter .....	.....	.....	.....
percent organic carbon .....	.....	.....	.....
carbonate as CaCO <sub>3</sub> .....	.....	.....	.....
percent insoluble carbonates .....	.....	.....	.....
pH (1:1 H <sub>2</sub> O) .....	.....	.....	.....
Cation exchange cap. (MEQ/100 g) .....	.....	.....	.....
Extractable cations (MEQ/100 g)			
Ca .....	.....	.....	.....
Mg .....	.....	.....	.....
Na .....	.....	.....	.....
K .....	.....	.....	.....
H .....	.....	.....	.....

Special Chemical/Mineralogical Features:

Clay Fraction Mineralogy:

**B. Results of preliminary test**

Test substance: \_\_\_\_\_

Sample purity: \_\_\_\_\_

Saturation concentrations: \_\_\_\_\_

Concentration of the test solution (show calculation): \_\_\_\_\_

Description of a suitable analytical method:

### C. Results of screening test

#### 1. Adsorption

	Soil I		Soil II		Soil III	
temperature, °C .....	.....	.....	.....	.....	.....	.....
C control .....	.....	.....	.....	.....	.....	.....
C <sub>i</sub> .....	.....	.....	.....	.....	.....	.....
C <sub>e</sub> .....	.....	.....	.....	.....	.....	.....
x .....	.....	.....	.....	.....	.....	.....
m .....	.....	.....	.....	.....	.....	.....
x/m .....	.....	.....	.....	.....	.....	.....
G .....	.....	.....	.....	.....	.....	.....
V .....	.....	.....	.....	.....	.....	.....

#### 2. Desorption

	Soil I		Soil II		Soil III	
temperature, °C .....	.....	.....	.....	.....	.....	.....
C <sub>i</sub> .....	.....	.....	.....	.....	.....	.....
D .....	.....	.....	.....	.....	.....	.....
R .....	.....	.....	.....	.....	.....	.....

#### 3. Final data

	I	II	III
temperature t, °C .....	.....	.....	.....
initial concentration, C <sub>i</sub> .....	.....	.....	.....
percent of chemical adsorbed on adsorbent, A .....	.....	.....	.....
percent desorbed, D .....	.....	.....	.....
percent of the original quantity found in the control sample remaining on adsorbent after desorption, R .....	.....	.....	.....
adsorption coefficient, K' .....	.....	.....	.....
adsorption coefficient based on organic carbon content, K' <sub>oc</sub> .....	.....	.....	.....
final concentration after correction, C <sub>e</sub> .....	.....	.....	.....

**D. Results of advanced test**

**1. Adsorption kinetics**

Soil Type I:

Temperature: \_\_\_\_\_

Time for reaching  $C_e$  \_\_\_\_\_

time						
$C_i$ .....	.....	.....	.....	.....	.....	.....
$C_e$ .....	.....	.....	.....	.....	.....	.....

Soil Type II:

Temperature: \_\_\_\_\_

Time for reaching  $C_e$  \_\_\_\_\_

time						
$C_i$ .....	.....	.....	.....	.....	.....	.....
$C_e$ .....	.....	.....	.....	.....	.....	.....

Soil Type III:

Temperature: \_\_\_\_\_

Time for reaching  $C_e$  \_\_\_\_\_

time						
$C_i$ .....	.....	.....	.....	.....	.....	.....
$C_e$ .....	.....	.....	.....	.....	.....	.....

## 2. Adsorption Isotherms for each concentration ( $C_i$ )

	Soil				Soil		
	I	II	III		I	II	III
$C_i$ .....	.....	.....	.....		.....	.....	.....
$C_e$ .....	.....	.....	.....		.....	.....	.....
$x$ .....	.....	.....	.....		.....	.....	.....
$m$ .....	.....	.....	.....		.....	.....	.....
$x/m$ .....	.....	.....	.....		.....	.....	.....
$C_i$ .....	.....	.....	.....		.....	.....	.....
$C_e$ .....	.....	.....	.....		.....	.....	.....
$x$ .....	.....	.....	.....		.....	.....	.....
$m$ .....	.....	.....	.....		.....	.....	.....
$x/m$ .....	.....	.....	.....		.....	.....	.....
$C_i$ .....	.....	.....	.....		.....	.....	.....
$C_e$ .....	.....	.....	.....		.....	.....	.....
$x$ .....	.....	.....	.....		.....	.....	.....
$m$ .....	.....	.....	.....		.....	.....	.....
$x/m$ .....	.....	.....	.....		.....	.....	.....

(g) **References.** The following references should be consulted for additional background material on this test guideline.

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