Adsorption and desorption of [test compound] in [number] soils [or sediments]

|  |  |
| --- | --- |
| Report: | [Provide full citation. Provide the MRID (first) if the review is unilateral.] |
| Document No.: | [MRID xxxxxxxx] |
| Guideline: | OCSPP 835.1230 [If the study was conducted under a different guideline, state ‘Conducted by’ and provide the most relevant guideline(s) the study was conducted under. Then state ‘Reviewed by OCSPP 835.1230.’ If this review is multilateral, also provide the guideline numbers under which participating agencies are reviewing the study.] |
| Statements: | [Indicate whether the study was conducted in compliance with FIFRA GLP standards and whether signed and dated Data Confidentiality, GLP Compliance, Quality Assurance, and Authenticity Certification statements were provided. If the study was not conducted in compliance with FIFRA GLP standards, indicate how not or why not.] |
| Classification: | This study is [provide classification and very concise statement of any deficiencies that impacted the classification]. [If multiple classification terminologies are needed for multilateral reviews, list or tabulate them.] |
| PC Code: | [xxxxxx] |
| Reviewer: | [Provide final reviewer(s)’s name Signature:and title.] Date: [Type date of signature.] |

**Executive Summary**

In a batch equilibrium study, [identify number of soils/sediments and origin and range of pH, *e.g.*, one U.S. soil and two European soils (pH range of 5.2 - 7.3)] were used to measure sorption coefficients of [type of radiolabel(s)]-labeled [test compound] at [temperature±degree of variation]. Determined Freundlich Adsorption Coefficient (KF) values ranged from [value] to [value] (mean KF=[value]); KFOC values ranged from [value] to [value] (mean KFOC= [value]).[[1]](#footnote-1) The Freundlich Desorption Coefficients (KF-des) for desorption were [greater than/similar to] ([provide range, mean KF =[value]]) those obtained for the initial sorption measurement and a total of [percentage] the amount initially sorbed was desorbed. [Discuss implications of results, *e.g*., is sorption reversible?] Freundlich exponents corresponding to KF values ranged from [provide range] indicating that sorption [was/was not] linear, *i.e*. the sorption coefficient [was/was not] independent of the test item concentration. Adsorption coefficients (Kd) ranged from [value] to [value and units] and organic carbon normalized adsorption coefficients (KOC) ranged from [report range of values and units]. Table [number] summarizes the adsorption coefficients measured in the study. Table [number] summarizes the desorption coefficients measured in the study. [Report coefficients of variation for Kd, KOC, KF and KFOC values and discuss which would be a better predictor of sorption across soils. Discuss whether the concentration range examined was appropriate.]

The mass balance at the end of the study ranged from [value] to [value] percent. It was verified that equilibrium was achieved in test systems, and the study was carried out for [duration]. Sorption to test vessel walls [was/was not] observed [if there was sorption to vessel walls, describe how the loss to vessel walls was handled in calculations of sorption coefficients].

Results Synopsis:

Table 1. Summary of Adsorption/Desorption Results A

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| Soil(% OC, pH) | Regressed Kd(L/kg-soil) | Range of Kd (L/kg-soil) | Regressed KOC (L/kg-OC) | KF((L/kg-soil)-1/n) | 1/n | KFOC(L/kg-OC)-1/n) | Ceq Range (mg/L) |
| Value±SE | r2 | p-value | Value±SE | Value±SE | r2 | p-value | Value±SE | Value±SE |
| Adsorption |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Desorption |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Soil(%OC, pH) |  | ± |  |  |  |  | - |  |  | ± |  |  | ± |  |  |  |  | ± |  |  | ± |  |  | - |  |
| Abbreviations: SE = standard error of regression; NR = not reported; NA = not applicable.A Data were obtained from [location of data in study report] and data and results are in the attached [Excel workbook or sigma plot file] [name(s) of worksheets, if needed]. See Attachment 3 for equations used for calculations. |

**I. Material and Methods**

**A. Materials:**

**1. Test Material:** [[Type of radiolabel]-labeled[test compound]
Specific radioactivity: [value] MBq/mg

 Radiochemical purity: [percentage (HPLC, TLC)]

 Chemical purity: [percentage (HPLC)]

 Batch number: [value]

 Solubility in water: [value] mg/L at [#]oC [If pH-dependent, list available values at each study pH.]

 pKa: [value]

**2. Reference** [List the common name and batch number of each reference

**Compounds:** compound. Provide other chemical information in the structure table.]

**3. Soils/Sediment:** The study was conducted with [number] different soil types ([number of European soils and number of U.S. soils]) and [number] of sediments ([number of European soils and number of U.S. sediments]). These soils were collected from the top [depth of soil layer in cm] layer in fields that had not been treated with a pesticide for at least [number] of years. Air dried soils were stored at ambient temperatures for [number] weeks prior to experimentation. A summary of the physical and chemical properties of the soils is provided in Table [identify table] [include USDA Soil Taxonomy and/or World Resources Base (WRB) soil classification]. [Indicate whether the soil is representative of use sites.]

**Table 2. Description of Soil/Sediment**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Soil NameOrigin |  |  |  |  |  |
| USDA Textural Class |  |  |  |  |  |
| % Sand |  |  |  |  |  |
| % Silt |  |  |  |  |  |
| % Clay |  |  |  |  |  |
| %OC1 |  |  |  |  |  |
| CEC2 |  |  |  |  |  |
| pH3 |  |  |  |  |  |
| % moisture (1/3 bar) |  |  |  |  |  |
| Soil Taxonomy |  |  |  |  |  |
| 1 %OM/1.72 = %OC. [Specify method used to measure OC.]2 [Specify method used to measure CEC.]3 Measured in a xx:xx soil water suspension. [Specify method used to measure pH.] |

**B. Study design:** [Tabulation of these data is encouraged as long as the length of this section is not substantially increased.]

1. Experimental conditions:

[Describe the preliminary studies conducted to determine the soil:solution ratio, equilibration period, sorption of test compound to the test vessels, stability of the test compound under test conditions, *etc.*]

Stock solutions of [type of radiolabel(s) test compound] in [solvent] were prepared and aliquots added to portions of 0.01 M CaCL2 solution to give a concentration range of [range of initial concentrations] mg/L, ensuring that the concentration of [solvent] in aqueous solution did not exceed 0.1 % by volume. The appropriate solution to soil ratio was determined in preliminary testing at [ratio; *e.g.*, 2 : 1 (*circa* 10 % sorption)]. Portions of test solution [volume] were shaken at [temperature] with samples of test soil ([value] g dry weight) for a [duration] equilibration period in darkness. Duplicate test systems were performed at each test concentration. Following centrifugation (x rpm for y minutes), the supernatant was decanted and aliquots were prepared for radioassay. Blank controls (solution +soil/sediment without test substance) were subject to the same test procedures to identify interfering compounds or contaminated soils/sediment. Blank controls (solution + test substance without soil/sediment) were subject to the same test procedures to assess potential sorption to glass test vessels and potential of loss due to instability. Following the sorption phase, fresh 0.01 M aqueous CaCL2 [volume] was added to each test vessel, equilibrated for [duration] at [temperature], solutions and soils separated, quantified and subject to a further desorption phase. Soil extracts from the highest concentration tested were further extracted by shaking twice with [number] ml [solvent]. The extracts were used to assess the degree of degradation of [test compound] during equilibration. Results [were/were not] corrected for the degradation observed.

[Describe soil collection and preparation, including sieve size and sterilization technique (if used).]

[Indicate other studies supporting the chemical registration that use the same soil.]

1. **Analytical procedures:** Radioactivity was determined by Liquid Scintillation Counting (LSC), and both aqueous supernatants and soil extracts obtained after equilibration were analyzed by [identify method of analysis] of the highest test concentration [identify concentration] samples. [Identify methods used to confirm chemical identity] were used to confirm the identity and concentrations of the parent and degradation products detected. The limit of detection (LOD) for [test substance and any metabolites] was [value(s)] μg as/g soil. The limit of quantification (LOQ) for [test substance and any metabolites] was [value(s)] μg/g soil.

**II. Results and Discussion**

**A. Mass Balance:** Recovery of radioactivity in aqueous supernatant and soil extracts on [describe when balance was taken and what extracts were used] ranged from [range of values] of the applied amount. Recoveries following desorption ranged from [range of values]. [Indicate whether there was substantial loss of radioactivity by sorption to glassware or volatilization. Indicate whether unidentified radioactivity occurred at levels that would impact study results.]

**B. Transformation of Parent Compound:** During the [duration] equilibration period, [test substance] was degraded to varying degrees depending on the soil/sediment. The major degradation product(s) was [metabolite X], which represented [range of values] % of the applied radioactivity in the soil extracts and [range of values] % in the aqueous supernatant. Each of the other degradation products accounted for <[value]% of the applied radioactivity.

**C.** **Findings:**

[Reported values were calculated using [identify program used to calculate coefficient] and the equations and methods discussed in the calculations section. Refer to tabulated adsorption and desorption coefficients. Discuss whether isotherms are linear or nonlinear. Report the range of adsorption and desorption coefficients. Regress Kd by percent organic carbon, CEC, and percent clay. Report the r2 and p-value for the regressions in a table. Include the graphs in the study review (see example below). Calculate coefficients of variation (*e.g*., standard deviation divided by the mean) across soils for Kd, KOC, KF and KFOC. If the coefficient of variation (CV) for KFOC is less than the CV for KF, then KFOC is a better predictor of sorption across soils. Discuss whether evidence indicates that organic carbon, CEC, and pH influence sorption. Note whether the relationships were confounded by factors such as pH and pKa, factors not being held constant across soils, *etc*. Compare the range of desorption coefficients with the range of adsorption coefficients for the same soil/sediment. Provide the range of percentages of the amount of sorbed that was desorbed. Provide the range of equilibrium concentrations in water and discuss whether the ranges were appropriate. Discuss whether sorption to the walls of the test system or instability of test compound influenced the results. Report the pH of the equilibrium solution. If the compound has a pKa and may be present in multiple association states, discuss whether sorption coefficients were measured over a range of environmentally relevant pH values and how pH may have influenced the results.]

[Indicate whether reviewer-reported sorption coefficients are consistent with study-reported values.]

[Include graphs of Ceq versus Cs for each isotherm. An example graph is provided below, as well as an example graph for Kd versus percent organic carbon.]

**III. Study Deficiencies and Reviewer’s Comments**

[This section is titled “Conclusions” in the original T2S template.]

[List any deficiencies with the study and any additional salient information. Results and conclusions contained in the Executive Summary are not repeated in this section.]

**IV. References**

[List any references cited in the review.]

**Attachment 1: Chemical Names and Structures**

**[**A table (*i.e.*, structure table) of the chemical names, SMILES strings, CAS numbers, and structures of the test compound, identified transformation products, and reference compounds that were not identified in study samples should be either referenced as a separate, associated document or attached to the study review. Multiple versions of structures to show or not show radiolabeling and multiple versions of chemical names and SMILES strings should not be included in the table. Sources of data need not be included. However, formatting the structure table in conformance with the guidance for tabulating transformation product data for EFED ROCKS memoranda is recommended. This formatting includes table columns for MRIDs and associated study data such as maximum and final concentrations of transformation products and their intervals. At a minimum, repeat the table below for the test compound, identified transformation products, and reference compounds not identified in the study samples.

For multilateral reviews, chemical names, SMILES strings, structures, and CAS numbers are captured elsewhere in the Monograph. Therefore these data are not attached to each study review within the Monograph. When the Monograph is split into individual reviews in EFED’s files, however, the Monograph’s structure table should be either referenced as a separate, associated document or attached to each individual review.]

[Sample structure table with the minimum information needed.]

|  |
| --- |
| **[Common name [list other common names] [if the same common name is used in different studies for different compounds, provide in parentheses the MRID associated with the common name for this compound.]]** |
|  |  |
| IUPAC Name: | [Provide one IUPAC name.] |
| CAS Name: | [Provide one CAS name.] |
| CAS Number: | [Provide if available.] |
| SMILES String: | [Provide one SMILES string.] |
|  |
| [Paste structure here.] |
|  |
|  |

[Sample EFED ROCKS memorandum format for structure tables.]



Attachment 2: Statistics Spreadsheets and Graphs

[Supporting electronic spreadsheet files should be inserted here (electronic attachment files should be electronically finalized as separate files as well). Electronic attachments should have the same file name as the Microsoft Word study review file with the addition of “Calc” for Excel workbooks and WinZip files, the addition of “Data” for Adobe Acrobat and Document Imaging files, and the addition of brief descriptors as appropriate for SigmaPlot Notebooks. Electronic attachment files should be compressed into a WinZip file when three or more are prepared for a study review.]

[Hard copies of a study review and any attachment sheets from separate electronic files should be printed and finalized together as one hard copy file.]

**Attachment 3: Calculations**

Calculations were performed by the reviewer using [indicate program(s) used for calculations] and the following equations.

Ceq range is the range of [test substance] concentrations in water at equilibrium.

Cs is the concentration of [test substance] sorbed to soil or sediment at equilibrium.

Kd –Distribution Coefficient for Adsorption = Cs/Ceq (eq 1)

* Regressed Kd is calculated using linear regression of Cs versus Ceq with a forced zero intercept over the range of measured Ceq for each soil/sediment.
* Range of Kd reflects the range of each Kd measured at a specific concentration in a soil/sediment

KOC - Organic Carbon Normalized Adsorption Coefficient = regressed Kd \*100/% OC (eq 2)

Standard Error (SE) of KOC= Kd SE \*100/%OC (eq 3)

KF - Freundlich Adsorption Coefficient and the Freundlich exponent (1/n) were calculated using nonlinear regression of Cs = KF x Ceq 1/n. Cs should be expressed in mg/kg and Ceq should be expressed in mg/L in the regression. (eq 4)

KFOC –Organic Carbon Normalized Adsorption Coefficient = KF\*100/%OC (eq 5)

Standard Error (SE) of KFOC= KF SE \*100/%OC (eq 6)

KDES –Apparent Desorption Coefficient = Cs/Ceq where Cs and Ceq are measured after an initial sorption measurement and the soil/sediment is placed in a new solution and allowed to equilibrate, so that any material in solution desorbed from the soil/sediment. (eq 7)

* Regressed KDES is calculated using linear regression of Cs versus Ceq over the range of Ceq measured with a forced zero intercept for each soil/sediment.
* Range of KDES reflects the range of each KDES measured at a specific concentration in a soil/sediment

KOC-DES - Organic Carbon Normalized Apparent Desorption Coefficient = regressed KDES \*100/% OC (eq 8)

Standard Error of Koc-DES = KDES SE\* 100/%OC (eq 9)

KF-DES - Freundlich Desorption Coefficient and the Freundlich Desorption exponent (1/n) were calculated using nonlinear regression of Cs = KF-DES x Ceq 1/n (eq 10)

KFOC-DES - Organic Carbon Normalized Freundlich Desorption Coefficient= KF-DES\*100/%OC (eq 11)

Standard Error of KFOC-DES = KF SE \*100/%OC (eq 12)

1. Sorption is a generic term that applies to absorption, adsorption, and desorption processes. Adsorption refers to sorption onto a two-dimensional surface; absorption refers to sorption into a three dimensional matrix. Both types of sorption occur in soils and sediments. Desorption refers to a sorbate becoming desorbed from a sorbent. Desorption distribution coefficients are measured by removing solution from a sorption experiment and adding fresh solution, so that all material measured in solution will be the desorbed material. The guideline uses the term adsorption in place of sorption and refers to the initial measurement of sorption. [↑](#footnote-ref-1)