Laboratory volatility of [test compound]

|  |  |
| --- | --- |
| Report: | [Provide full citation. Provide the MRID (first) if the review is unilateral.] |
| Document No.: | [MRID xxxxxxxx] |
| Guideline: | OCSPP 835.1410  [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 835.1410.’ 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 why or how it deviated.] |
| 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 laboratory study, the volatility of [test compound] was investigated on [soil texture] under aerobic soil conditions at [x] ± [x] ºC for a period of [x] days. Based on the similarities/ differences in the World Resources Base (WRB) soil classification of the study soil and the use sites, the soil used [is/is not] representative of an intended use-site. Soil samples were treated at [date:time] at [test concentration] mg a.i./kg, which is equivalent to a field application rate of [#] g a.i./ha ([#] lbs a.i./a). Two replicates for the treated test system were examined in the study. [Include brief sentence about how air concentrations were determined, if a soil analysis was conducted, and if unextracted residues were analyzed.] Two replicates were collected and analyzed using [describe extraction and analytical methods used (*e.g.*, LSC and HPLC-UV)].

The volatility profile of [test compound] was [add a brief description to explain the volatility pattern]. The test system [was/was not] adequate to trap the parent and its transformation products. Major transformation products included [x], [y] and [z]. The maximum observed air concentration of [test compound] was [#] µg/m3 during Sampling Period [#], [#] hours after application. The maximum estimated volatility (*e.g.*, flux rate) of [test compound] was [#] µg/m2-s during Sampling Period [#], [#] hours after application. At the end of the study, [#] percent of the amount applied had been volatilized. The air concentrations and volatility estimates for the various sampling periods are provided in **Table 5**.

The duration of the experiment was [not adequate/adequate] to see the decline in air concentrations of the parent and transformation products.

**I. Material and Methods**

Provide (a) small image(s) of the active ingredient(s) in the right margin.

1. **Materials**

**1. Test Material**

Table 1. Properties of Test Material

| Property | Value |
| --- | --- |
| Product Name |  |
| Formulation Type | [*e.g.*, liquid or granular] |
| Typical end-use product? | [Yes/No] |
| Contaminants and/or impurities |  |
| Manufacture # |  |
| Lot # |  |
| [[Type of radiolabel]-labeled |  |
| Specific radioactivity | [value] [units] |
| Radiochemical purity | [percentage [HPLC or TLC] |
| CAS # |  |
| Chemical structure | [Provide (a) small image(s) of the test compound(s) structure and any radiolabel.] |
| Storage stability | [Indicate the time period that the test material was stable as well as any degradation rate information.] |
| pH |  |

**2. Storage Conditions**

[Indicate if the test material was frozen, refrigerated, and/or maintained in the dark for [#] days.]

**3. Soil**

Table 2. Soil(s) Collection, Storage and Properties

| Property | Value |
| --- | --- |
| Geographic location |  |
| Pesticide use history at the collection site |  |
| Collection date |  |
| Collection procedures |  |
| Sampling depth |  |
| Storage conditions |  |
| Storage duration |  |
| Soil preparation |  |
| Soil texture (USDA[[1]](#footnote-1)): |  |
| % Sand |  |
| % Silt |  |
| % Clay |  |
| pH (1:1 soil:water or other)  [method] |  |
| Organic carbon (%)  [method] |  |
| Organic matter (%)  [method] |  |
| CEC (meq/100 g)  [method] |  |
| Soil Moisture Content (units): |  |
| At 0.1 bar (pF 2.0) |  |
| At 1/3 bar (pF 2.5) |  |
| Bulk density(g/cm3) |  |
| Microbial biomass (units): |  |
| At initiation |  |
| At termination |  |
| Soil taxonomic classification (WRB) |  |

Data obtained from pages [#] of the study report.

[Add columns if multiple soils are assessed.]

1. **Study Design**
2. Experimental Conditions

[Briefly describe the setup of the lab volatility equipment and how environmental conditions (*e.g.*, temperature, relative humidity, soil moisture, etc.) are to be maintained and monitored.]

Table 3. Experimental Design

| **Parameter** |  | Description |
| --- | --- | --- |
| Duration of the test (days) | |  |
| Soil condition (Air dried/fresh) | |  |
| Soil sample weight (g/replicate) | |  |
| Soil depth (cm) |  |  |
| Test concentration (mg ai/kg soil (dry weight)) | |  |
| Field Equivalent Application Rate (lb a.i./A) | |  |
| Number of replicates |  |  |
| Test apparatus |  |  |
| Test material application | Test solution volume used/ treatment |  |
|  | Application method |  |
| Indication of test material adsorbing to walls of test apparatus? | |  |
| Experimental conditions | Temperature (°C) |  |
|  | Relative humidity |  |
|  | Soil moisture content |  |
|  | Moisture maintenance method |  |
|  | Air flow through system |  |
|  | Continuous darkness (Yes/No): |  |
| Other observations (if applicable) | |  |

Data obtained from pages [#] of the study report.

1. Sampling during Study Period

[Briefly describe the air sampling equipment and how air samples were collected. Also describe any soil samples that may have been collected.]

Table 4. Sampling Design

| **Parameter** |  | Description |
| --- | --- | --- |
| Air Sampling | | |
| Sample intervals (units) |  |  |
| Sampling method | |  |
| Desired air flow of sampler (L/min) | |  |
| Sample storage before analysis (Yes/No)? | |  |
| Soil Sampling | | |
| Sample intervals (units) | |  |
| Sampling method | |  |
| Sample storage before analysis (Yes/No)? | |  |

Data obtained from pages [#] of the study report.

1. Sample Handling and Storage Stability

[Provide a brief description of the storage conditions of samples after collection and the longest duration of storage for the air and soil samples analyzed. Indicate the stability of the analytes based on the submitted storage stability study(ies) (provide MRID(s)) and whether the storage stability study duration(s) was/were sufficient to evaluate the longest storage duration(s). State whether corrections were made to account for any instability. Details of the storage stability study(ies) can be discussed in a separate study review(s).]

1. Analytical Procedures

Extraction methods: [Briefly describe the extraction method for the sorbent material and the soil, if soil was analyzed. Include information regarding the retrieval of parent and transformation products. Be sure to describe the effectiveness of the extraction process. Solvent selection should be sufficiently robust to ensure extraction from the sorbent and the soil. An example would be the following:

*Sorbent samples were extracted [# of extractions] with [solvent system] (%:%, v:v) by [extraction method] for [#] minutes per extraction ([report page reference]). Extracts were combined, diluted with [solvent], and pH adjusted [pH value] using [dilution solvent]. Aliquots of the combined extracts were concentrated under a stream of [gas] in a waterbath, diluted with [dilution solvent] and filtered (# μm and type of filter). Aliquots of the concentrated solutions were analyzed by [analytical method].*]

*Soil samples were extracted [# of extractions] with [solvent system] (%:%, v:v) by [extraction method] for [#] minutes per extraction ([report page reference]). After each soil extraction, the mixtures were centrifuged and the supernatant decanted. Extracts were combined, diluted with [solvent], and pH adjusted [pH value] using [dilution solvent]. Aliquots of the combined extracts were concentrated under a stream of [gas] in a waterbath, diluted with [dilution solvent] and filtered (# μm and type of filter). Aliquots of the concentrated solutions were analyzed by [analytical method].*]

Total Radioactivity Measurement: Total 14C residues were determined by summing the percent of applied mass found in residues measured in the soil extracts and air samples ([report page number]). Radiolabeled [parent compound] was identified by comparison to the retention time of an unlabelled reference standard (purity [%], retention time (Rt) ([# to #] minutes) that was co-chromatographed with the samples ([report page number]). Column recovery determined prior to analysis of the definitive samples was [#] % ([report page number]).

Identification and Quantification of Parent Compound: [Briefly describe how the extracts from the sorbent material were analyzed for the parent and transformation products. An example would be: *Aliquots of the sorbent extracts were analyzed using [analytical method (i.e., HPLC)] under the following conditions: [describe instrument, column, mobile phase, gradient, and UV (# nm) ([report page number]). The identification of [parent compound] was confirmed by [analytical method (i.e., LC-MS/MS)] with electrospray ionization in the positive mode ([report page number]).*]

Detection Limits (LOD, LOQ) for the Parent Compound: The limit of detection (LOD) was determined to be [#] μg, or [percentage]% of the applied radioactivity (%AR), with a limit of quantitation (LOQ) at [#] μg, or [percentage]% AR. The method for determining the LOD and LOQ was/was not adequate. [Include information for all analytical methods used in study.]

Detection Limits (LOD, LOQ) for the Transformation Products: The LOD for the transformation products was determined to be [#] μg, or [percentage]% of the applied radioactivity (%AR), with an LOQ at [#] μg, or [percentage]% AR. The method for determining the LOD and LOQ was/was not adequate. [Include information for all analytical methods used in study. If the LOD and LOQ were the same as parent, then cite same].

Instrument performance: [Include general information on method used for instrument calibration.] *A calibration curve was created using 5 calibration standards and spanning XX concentrations and response range.*

Lab recovery, air sampling sorbent material: [Most/All] laboratory spike recoveries are within the acceptable range with overall recoveries between [x and y percent]. The exceptions include laboratory spikes extracted concurrent with [sampling period] with an average percent recovery of [x percent + y percent] at the fortification level of [<c1 μg, c1 μg – c2 μg, or c2 μg>]. [Repeat for additional unacceptable laboratory spike extractions (less than 90 percent or greater than 110 percent).]

Lab recovery, soils: [Most/All] laboratory spike recoveries are within the acceptable range with overall recoveries between [x and y percent]. The exceptions include laboratory spikes extracted concurrent with [sampling period] with an average percent recovery of [x percent + y percent] at the fortification level of [<c1 μg, c1 μg – c2 μg, or c2 μg>]. [Repeat for additional unacceptable laboratory spike extractions (less than 90 percent or greater than 110 percent).]

Breakthrough, air samples: [Describe any analysis or instances of test substance breakthrough in sorbent material, if applicable.]

**II. Results and Discussion**

1. **Data**

Sample durations, sample volume flowrates, and the mass of the parent and transformation products collected on the sorbent material are shown in **Table 5**. Air concentrations were derived by dividing the mass of the parent by product of the sample volume flowrate and sample duration. Air concentrations for the parent ranged from [#] to [#] μg/m3.

1. **Material Balance**

The material balance and the distribution of radioactivity in the air and soil samples are shown in **Table 4**. Recoveries ranged from [percentage]% to [percentage]% of the applied radioactivity (%AR). Unidentified residues accounted for [percentage]% AR. [If there is a large amount of unidentified radioactivity, mention it here. Indicate whether there was substantial loss of radioactivity by sorption to glassware. Also mention if the mass balance meets guideline criteria.]

1. **Study Conditions**

Soil moisture, temperature, and microbial biomass [were/were not] maintained throughout the study.

1. **Transformation Products**

[Briefly summarize the air concentrations of the transformation products in **Table 5.**]

1. **Volatilization**

Rates of volatilization (*e.g.*, flux) for the parent are provided in **Table 5**. Volatilization flux rates were estimated by multiplying the air concentration (μg/m3) by the system ventilation flowrate (m3/s) and dividing by the horizontal surface area of the soil sample (m2). [Note: if this is a closed system (*e.g.*, air is pulled exclusively through the sorbent tube before exiting the system), the system ventilation rate will be the same as the sampler flowrate. If the measured percent recovery for the extraction method is not consistent across extractions, the air concentrations should be corrected based on the percent recovery for the extraction procedure.] The maximum volatilization rate for the parent in viable soil was [#] μg/m2-s at sample period [#] and declined to [#] μg/m2-s at sample period [#]. The maximum volatilization rate for the parent in sterilized soil was [#] μg/m2-s at sample period [#] and declined to [#] μg/m2-s at sample period [#].

Table 5. Volatility of [test compound] and its transformation products from soil [Add to the table as needed for additional products. The results of mass and air concentrations should be presented as individual replicate values rather than means and standard deviations.]

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Period | | 1 | | 2 | | 3 | | 4 | | 5 | | n | | Total |
| Hours After Treatment | | # | | # | | # | | # | | # | | # | |
| Air Concentration Analyses | | | | | | | | | | | | | | |
| Sample Duration (hours) | |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Sample Volume Flowrate (m3/s) | |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Parent | Collected mass (µg) |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Air concentration (µg/m3) |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Volatility (µg/m2-s) |  |  |  |  |  |  |  |  |  |  |  |  |  |
| % of applied radioactivity |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Transformation Product 1 | Collected mass (µg) |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Air concentration (µg/m3) |  |  |  |  |  |  |  |  |  |  |  |  |  |
| % of applied radioactivity |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Transformation Product 2 | Collected mass (µg) |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Air concentration (µg/m3) |  |  |  |  |  |  |  |  |  |  |  |  |  |
| % of applied radioactivity |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Soil Analyses | | | | | | | | | | | | | | |
| Parent | | Soil concentration (µg/kg) | | | | | | | | | |  |  |  |
| % of applied radioactivity | | | | | | | | | |  |  |  |
| Transformation Product 1 | | Soil concentration (µg/kg) | | | | | | | | | |  |  |  |
| % of applied radioactivity | | | | | | | | | |  |  |  |
| Transformation Product 2 | | Soil concentration (µg/kg) | | | | | | | | | |  |  |  |
| % of applied radioactivity | | | | | | | | | |  |  |  |
| Unextracted residues | | % of applied radioactivity | | | | | | | | | |  |  |  |
| Total | | % of applied radioactivity | | | | | | | | | |  |  |  |

Data obtained from pages [#] of the study report. Data should not be entered in cells that are gray.

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

[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**

[Attach a table (*i.e.*, structure table) of the chemical names, SMILES strings, CAS numbers, and structures of the analytes (*i.e.*, the test compound, identified transformation products, and reference compounds that were not identified in study samples) or refer to this table if it exists in a separate, associated document. Do not include in the table multiple versions of chemical names and SMILES strings. 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 analytes.

For multilateral reviews, chemical names, SMILES strings, structures, and CAS numbers are captured elsewhere in the Monograph[[2]](#footnote-2). 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, either reference the Monograph’s structure table as a separate, associated document or attach it 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



[Insert supporting electronic spreadsheet files here (electronic attachment files are electronically finalized as separate files as well). Name electronic attachments 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. Compress electronic attachment files into a WinZip file when three or more are prepared for a study review.]

[Print hard copies of the study review and any attachment sheets from separate electronic files to produce one hard copy file for finalization.]

Calculations used in the spreadsheet are summarized below.

where

C = Air concentration (µg/m3)

M = Collected mass (µg)

F = Sample volume flowrate (m3/s)

D = Sample duration (hours)

CF = Conversion factor (3600 s/hour)

where

V = Volatility (flux) (µg/m2-s)

M = Collected mass (µg)

A = Soil horizontal surface area of (m2)

D = Sample duration (hours)

CF = Conversion factor (3600 s/hour)

where

Pct = Percent of applied radioactivity (%)

V = Volatility (flux) (µg/m2-s)

D = Sample duration (hours)

AR = Application rate (lbs ai/A)

CF = Conversion factor (0.0321 s-lb-m2/µg-A [3600 s/hour x 1x10-6 g/µg x 0.002205 lbs/g x 4046.86 m2/A])

1. U.S. Department of Agriculture [↑](#footnote-ref-1)
2. A Monograph is a collection of multiple study reviews and data summaries prepared by government agencies into a single document that follows an OECD format. Typically, Tier II Summaries prepared by industry are updated by government agencies based on agency-review and then placed within the Monograph. [↑](#footnote-ref-2)