UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF CHEMICAL SAFETY
AND POLLUTION PREVENTION

DEC 2 0 2012

MEMORANDUM

SUBJECT: **Additional Guidance for Reviewing Environmental Fate Studies**

FROM: Donald J. Brady, Ph.D., Director
Environmental Fate and Effects Division (7507P)
Office of Pesticide Programs

TO: Environmental Fate and Effects Division (7507P)
Office of Pesticide Programs

Through issuance of this memorandum, I am providing guidance to the Environmental Fate and Effects Division (EFED) on the review of a second set of environmental fate and related studies conducted under Office of Chemical Safety and Pollution Prevention (OCSPP) guidelines.[1] EFED reviews the subject studies in support of drinking water exposure and ecological risk assessments. The attached six study review guides (Attachments 1-6) are intended to help EFED scientists uniformly consider the issues, data, and formatting that are relevant to efficiently review these environmental fate studies. This guidance is effective immediately and supersedes the acceptance criteria for these studies provided in the 1989 FIFRA Accelerated Reregistration Phase 3 Technical Guidance. This guidance also provides new DER templates for use in the interim while a NAFTA-harmonized DER format is negotiated with the NAFTA partners.

Attachments

Attachment 1: Laboratory Volatility Study Review Guide
Attachment 2: Field Volatility Study Review Guide
Attachment 3: Fish and Oyster BCF Study Review Guide
Attachment 4: Sample Storage Stability Study Review Guide
Attachment 5: Prospective Ground Water Monitoring Study Review Guide
Attachment 6: Environmental Chemistry Method (ECM) and Independent Laboratory Validation (ILV) Report Review Guide

[1] OCSPP guidelines are also referred to as OPPTS guidelines because the guidelines have not been updated following the Office of Prevention, Pesticides, and Toxic Substances' (OPPTS) name change to the Office of Chemical Safety and Pollution Prevention (OCSPP). Therefore, the term 'OPPTS' remains in the guidelines' text.

Laboratory Volatility Study Review Guide

This guide was developed to aid in the review of laboratory volatility studies submitted under the Office of Chemical Safety and Pollution Prevention (OCSPP) guideline 835.1410. While the Organisation for Economic Co-operation and Development (OECD) does not have currently a Tier II Summary (T2S)[1] specifically for laboratory volatility study reviews, the attached format is based on the format for other OECD T2S. Reviewers should add to the format any information, statistical calculations, and formating that will increase their utility to the Agency under OCSPP guideline 835.1410. In developing the study reviews, reviewers should strive to write concisely and to minimize alterations to the format.

Sections I, II, and III of this document provide details regarding formatting, data, and issues to consider in developing OCSPP environmental fate study reviews. Section I identifies data and review formatting that should be included in laboratory volatility reviews. Although this guide does not strictly prescribe where and how to present the data, an example study review template is provided in Section II for guidance. In general, reviewers should follow the example template, especially for unilateral reviews (*i.e.*, reviews conducted without the participation of other agencies). However, reviewers may modify the template as needed or disregard it in the case of multilateral reviews (*i.e.*, reviews with other agencies participating) in which an alternative format is agreed upon by the participating agencies. Lastly, a list of review considerations (Section III) is provided to help reviewers focus on critical study issues and to identify any common major or minor deficiencies (*i.e.*, the review considerations update the acceptance criteria for laboratory volatility studies provided in the 1989 FIFRA Accelerated Reregistration Phase 3 Technical Guidance (USEPA, 1989)).

References

U.S. Environmental Protection Agency (USEPA). 1989. FIFRA Accelerated Reregistration, Phase 3 Technical Guidance. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 540/09-90-078.

[1] A Tier II Summary is an OECD format for study reviews prepared by industry. See OECD Guidance Documents for Pesticide Registration at http://www.oecd.org/chemicalsafety/agriculturalpesticidesandbiocides/oecdguidancedocumentsforpesticideregistration.htm (Accessed Dec. 12, 2012).

Section I. Data Considerations for Laboratory Volatility Study Design

- The Guidelines require the use of a typical end-use product (TEP), but different formulations will affect volatilization. The TEP used in the study should be selected based on the anticipated outcome of the study. Is the study trying to evaluate how the TEP formulation reduces volatile emissions, or is it trying to estimate upper-end volatility?

- The duration of the study should be sufficient to see a decrease in volatile emissions (*e.g.*, flux rates). For highly volatile active ingredients, emissions will increase and then decrease over a short period of time (*e.g.*, less than a few days). However, for semi-volatile chemicals (e.g., chemicals with a vapor pressure between 10^{-4} and 10^{-6} torr), emissions may increase and then decrease over the course of several weeks.

- Soil moisture can impact the results of the study. Typically, if soil moisture is low, chemicals will volatilize more rapidly. However, the volatilization of some chemicals will increase with soil moisture due to displacement. The physical and chemical properties of the active ingredient should be carefully assessed. It may be preferable to assess both dry soil, with sufficient moisture to still maintain microbial activity, and moist soil, with a moisture content of 60% of the soil's water holding capacity (WHC).

- A sandy soil is recommended for use in this study type, as the impact of soil sorption will be minimized. If a different soil texture is used, the reviewer should note any measures that were taken to avoid reductions in volatility due to sorption to soil and the rationale for using a soil other than sand.

- Submission of a protocol prior to the conduct and submission of the laboratory volatility study is recommended. The protocol's Study Director should provide a justification for the particulars of the study design, which should be taken into account when reviewing the study and identifying deviations.

Section II. Example Laboratory Volatility Study Review Template

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 and title.] **Signature:** **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/m^3 during Sampling Period [#], [#] hours after application. The maximum estimated volatility (*e.g.*, flux rate) of [test compound] was [#] µg/m^2-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

A. Materials

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

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[2]):	

[2] U.S. Department of Agriculture

Property	Value
% 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/cm^3)	
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.]

B. Study Design

1. 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	

Parameter		Description
	Moisture maintenance method	
	Air flow through system	
	Continuous darkness (Yes/No):	
Other observations (if applicable)		

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

2. 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.

3. 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).]

4. 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 ^{14}C 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 $\pm$ 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

A. 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/m^3.

B. 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.]

C. Study Conditions

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

D. Transformation Products

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

E. 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/m^3) by the system ventilation flowrate (m^3/s) and dividing by the horizontal surface area of the soil sample (m^2). [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 [#] $\mu g/m^2$-s at sample period [#] and declined to [#] $\mu g/m^2$-s at sample period [#]. The maximum volatilization rate for the parent in sterilized soil was [#] $\mu g/m^2$-s at sample period [#] and declined to [#] $\mu g/m^2$-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 (m^3/s)														
Parent	**Collected mass (µg)**													
	Air concentration (µg/m^3)													
	Volatility (µg/m^2-s)													
	% of applied radioactivity													
Transformation Product 1	**Collected mass (µg)**													
	Air concentration (µg/m^3)													
	% of applied radioactivity													
Transformation Product 2	**Collected mass (µg)**													
	Air concentration (µg/m^3)													
	% 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[3]. 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.]

Degradate Table.docx

[3] 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.

Attachment 2: Statistics Spreadsheets and Graphs

Laboratory Volatility Study Review Spread

[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.

$$\frac{M \times F \times CF}{D}$$

where

C	=	Air concentration ($\mu g/m^3$)
M	=	Collected mass (μg)
F	=	Sample volume flowrate (m^3/s)
D	=	Sample duration (hours)
CF	=	Conversion factor (3600 s/hour)

$$\frac{M \times CF}{A \times D}$$

where

V	=	Volatility (flux) ($\mu g/m^2$-s)
M	=	Collected mass (μg)
A	=	Soil horizontal surface area of (m^2)
D	=	Sample duration (hours)
CF	=	Conversion factor (3600 s/hour)

$$\frac{V \times D \times AR \times CF}{100}$$

where

Pct	=	Percent of applied radioactivity (%)
V	=	Volatility (flux) ($\mu g/m^2$-s)
D	=	Sample duration (hours)
AR	=	Application rate (lbs ai/A)
CF	=	Conversion factor (0.0321 s-lb-$m^2/\mu g$-A [3600 s/hour x $1x10^{-6}$ g/μg x 0.002205 lbs/g x 4046.86 m^2/A])

Section III. Laboratory Volatility Study Review Considerations

This list of considerations is provided to help reviewers assess the acceptability of laboratory volatility studies. The list is not a screen or a checklist and is not meant to be attached to the study reviews. Listed considerations carry unequal weight. Evaluate them using best professional judgment. Consider all information from the study and from similar studies to determine whether any deficiencies affect the study classification.

Test Material

» The test compound was a typical end-use product (TEP). The composition of the TEP, including known contaminants and impurities, was provided.

» The TEP formulation was typical of the formulation category (*i.e.*, wettable powder, emulsifiable concentrate, *etc.*).

Soils

» A sandy soil, as recommended in OCSPP 835.4100 (laboratory volatility study), was used or measures were taken to avoid reductions in volatility from adsorption to soil.

» The soil's target moisture content was reported and adequately maintained during the course of the experiment.

» The soil was completely characterized, using the USDA or World Resources Base (WRB) soil classification system. If a foreign soil(s) was/were used, the soil(s) was/were adequately compared with domestic (U.S.) soils.

» The soil was sieved using a 2 mm screen.

Experimental Design

» The application rate was representative of the maximum intended use.

» Duplicate systems were sampled at each interval.

» The tests were conducted in darkness.

» The air temperatures were held at ±2°C between 18 and 30ºC, but 25ºC is preferred (the guideline does not specify requirements for an air temperature).

» The relative humidity and air movement in the test systems were adequately described and monitored.

» Air sampling was continuous or at a minimum of four sampling intervals. A soil sample was collected at least at the final interval. Sampling times were adequate to describe the flux rate over time and the decline curve of the parent and major degradates.

Analytical Design

» Raw measured data and representative chromatographs were provided.

» All relevant chromatographic peaks were quantified unless <LOQ. If there were interferences and the analysis was not conducted with a radiolabeled substance, supporting information was provided to indicate which of the peaks were and were not relevant.

» Limits of detection and quantification were reported and were adequate for the test substance.

» A reasonable attempt was made – perhaps with a polar and multiple mild non-polar solvent systems – to extract the test compound and its transformation products.

Report

» Air concentrations and volatilization rates were provided. Mathematical equations, used in generating and analyzing the data, were also provided.

» If rates of formation and decline of the parent compound, or its transformation products, were reported, data were expressed as the mass, concentrations, or percentages of parent applied. Tabular data and graphs for decline curves were submitted as well. If a radiolabeled TEP was used, a material balance was provided.

Field Volatility Study Review Guide

This guide is meant to aid in the review of field volatility studies submitted under the Office of Chemical Safety and Pollution Prevention (OCSPP) guideline 835.8100. The field volatility study review format is loosely based on the format of the Organisation for Economic Co-operation and Development (OECD) Tier II Summaries (T2S)[1], although an OECD T2S format specific to field volatility studies is not available currently. In developing study reviews, reviewers should strive to write concisely and to minimize alterations to the recommended format.

Sections I and II of this document provide details regarding data, formatting, and issues to consider in developing OCSPP environmental fate study reviews. Although this guide does not strictly prescribe where and how to present the data, an example study review template is provided in Section I for guidance. In general, reviewers should follow the example template, especially for unilateral reviews (*i.e.*, reviews conducted without the participation of other agencies). However, reviewers may modify the template as needed or disregard it in the case of multilateral reviews (*i.e.*, reviews with other agencies participating) in which an alternative format is agreed upon by the participating agencies. In Section II of this document, a list of review considerations is provided to help reviewers focus on critical study issues and to identify any common major or minor deficiencies.

1 A Tier II Summary is an OECD format for study reviews prepared by industry. See OECD Guidance Documents for Pesticide Registration at http://www.oecd.org/chemicalsafety/agriculturalpesticidesandbiocides/oecdguidancedocumentsforpesticideregistration.htm (Accessed Dec. 12, 2012).

Section I. Example Field Volatility Study Review Template

Field volatility of [test compound]

Report:	[Provide full citation. Provide the MRID (first) if the review is unilateral.]
Document No.:	[MRID xxxxxxxx]
Guideline:	OCSPP 835.8100 [If the study was conducted under a different guideline than it is being reviewed under, state 'Conducted by' and provide the most relevant guideline(s) the study was conducted under. Then state 'Reviewed by OCSPP 835.8100.' If in multilateral review, 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 and title.] **Signature:** **Date:** [Type date of signature.]

Executive Summary [Modify the Executive Summary if required, incorporating the following critical elements and level of detail.]

Field volatilization of [test material] was examined from [bare plots or cropped plots] at [number] of plots(s)/site(s) in [location(s), state(s), province(s)]. The site(s) where the studies were conducted were at [locations]. The experiment(s) in [site(s)] was/were conducted for [number] days. The nominal application rate[s] in [plots(s)/site(s)] were [value] lbs. a.i./A. The treated plots were [value] m apart, and the control plot was [value] m away from the treated plot. [Provide details on differences with application methods between plot(s)/site(s).]

Under field conditions at Site 1/Plot 1, [test material] a peak volatile flux rate of __ $\mu g/m^2 \cdot s$ of the applied was measured accounting for ___ % of the applied observed ___ to ___ hours post-application. By the end of the study, a total of ___ % of [test material] volatilized and was lost from the field. Other secondary peak volatile flux rates of __ $\mu g/m^2 \cdot s$, __ $\mu g/m^2 \cdot s$,….,and ___ $\mu g/m^2 \cdot s$, occurred at ___ to ___, ___ to ___, …, and ___ to ___ hours post-application, respectively. [State hypotheses for reasons of specific off-gasing events. Repeat information for additional sites, plots, or applications].

[Include a time series plot of flux rates from the spreadsheets in Attachment 2.]

I. Materials and Methods

A. Materials

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

1. Test Material

Product Name:
Formulation Type (*e.g.*, liquid or granular):
CAS #:
Storage stability: [Indicate the time period that the test material was stable as well as any degradation rate information.]

2. Storage Conditions

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

B. Study Design

1. Site Description

[Indicate geographic location (nearby city), number of treated fields, and corresponding field sizes and crop cover. Indicate field type (*e.g.*, bare soil or cropped), field dimensions, soil type (*e.g.*, textural and taxonomic classification), and information on past land and pesticide use on plot(s). Indicate distance of separation between plots. Indicate the terrain characteristics (*e.g.*, rolling or flat terrain characterized by mixed trees and open fields, mixed small vegetative cover and open fields, or complete coverage by agricultural land).]

2. Application Details

Application rate(s): [Describe the target application rate and actual measured application rate including the method used to verify the actual application rate for all of the fields and plots.]

Irrigation and Water Seal(s): [If irrigation water was used, describe the amount of water applied in inches and method used to apply water to the field before and throughout the study.]

Tarp Applications: [If tarps were used, describe the tarp material used (e.g., HDPE, LDPE, etc. and brand), the type of application (broadcast or bedded strip), the percentage of field covered, the dimensions/number of areas covered, and the time duration of the covering]

Application Equipment: [Describe all equipment used in the application of the test substance.]

Equipment Calibration

Procedures: [Describe the methods used to calibrate the equipment to apply the targeted test material.]

Application Regime: The application rates and methods used in the study are summarized in **Table 1**.

Table 1. Summary of application methods and rates for [test substance]

Field	Application Method	Time of Application (Date and Start Time)	Amount [Test Substance] Applied (lbs)	Area Treated (acres)	Calculated Application Rate (lb ai/acre)	Reported Application Rate (gal ai/acre)
1	[#]	[#]	[#]	[#]	[#]	[#]
2	[#]	[#]	[#]	[#]	[#]	[#]
3	[#]	[#]	[#]	[#]	[#]	[#]
n	[#]	[#]	[#]	[#]	[#]	[#]

Application Scheduling: Critical events of the study in relation to the application period are provided in **Table 2**.

Table 2. Summary of [test substance] application and monitoring schedule

Field/Plot	Treated Acres	Application Period	Initial Air/Flux Monitoring Period	Water Sealing Period	Tarp Covering Period
Field 1	[#]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY [##:##] – MM/DD/YY [##:##]
Field 2	[#]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY [##:##] – MM/DD/YY [##:##]
Field 3	[#]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY [##:##] – MM/DD/YY [##:##]
Field n	[#]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY between [##:##] – [##:##]	MM/DD/YY [##:##] – MM/DD/YY [##:##]

[If application methods other than water sealing or tarp coverings are included, please include this in **Table 2** as well.]

3. Soil Properties

Soil properties measured before the study are provided in **Table 3**.

Table 3. Summary of soil properties for fields/plots

Field	Sampling Depth	USDA Soil Textural Classification	USGS Soil Series	WRB Soil Taxonomic Classification	Bulk Density (g/cm^3)	Soil Composition
[#]	[#] – [#]	[#]	[#]	[#]	[#]	% Organic Carbon = [#]% % Sand = [#]% %Silt = [#]% %Clay = [#]%
[#]	[#] – [#]	[#]	[#]	[#]	[#]	% Organic Carbon = [#]% % Sand = [#]% %Silt = [#]% %Clay = [#]%
[#]	[#] – [#]	[#]	[#]	[#]	[#]	% Organic Carbon = [#]% % Sand = [#]% %Silt = [#]% %Clay = [#]%
[#]	[#] – [#]	[#]	[#]	[#]	[#]	% Organic Carbon = [#]% % Sand = [#]% %Silt = [#]% %Clay = [#]%

[Also include plots of soil temperature (°C) and soil moisture (% of field capacity) measured throughout the study.]

4. Meteorological Sampling

[Describe the meteorological instrumentation and vertical profile of measurements taken, if applicable. Details of the sensor heights and the meteorological parameters for which data were collected are illustrated in **Table 4**. The location of the meteorological equipment for each field is shown in **Attachment 3**.]

Table 4. Summary of meteorological parameters measured in the field

Field	Minimum Fetch* (m)	Parameter	Monitoring heights (m)	Averaging Period
1	[#]	Wind speed/Wind direction	Height [z_1], ….Height [z_n]	1 minute
		Ambient air temperature	Height [z_1], ….Height [z_n]	1 minute
2	[#]	Wind speed/Wind direction	Height [z_1], ….Height [z_n]	1 minute
		Ambient air temperature	Height [z_1], ….Height [z_n]	1 minute
		Solar radiation	Height [z_i]	15 minutes
		Precipitation	Height [z_i]	15 minutes
3	[#]	Wind speed/Wind direction	Height [z_1], ….Height [z_n]	1 minute
n	[#]	Ambient air temperature	Height [z_1], ….Height [z_n]	1 minute
		Ambient air temperature	Height [z_1], ….Height [z_n]	1 minute

*Only include for on-field flux air sampling. Values calculated in spreadsheets provided in Attachment 2.

5. Air Sampling

[Describe the air monitoring sampling network (*e.g.*, sampling mast for on-field monitoring and levels above ground for sampling or distribution of samplers around the treated field stations and

distances from the edge of the field). Also describe the pre-application monitoring (*e.g.*, placement of air samplers, total monitoring duration, and averaging periods for sampling).]

6. Sample Handling and Storage Stability

[Provide a brief description of the storage conditions of samples after collection and the longest duration of storage for each media analyzed. Indicate the stability of the analytes based on the submitted storage stability study(ies) for each media (provide MRID(s)) and whether the storage stability study duration(s) was/were sufficient to evaluate the longest field study 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).]

7. Analytical Methodology

[Describe the following attributes of the air sampling analysis:

- Sampling Procedure and Trapping Material:

  [Include apparatus, air pump flow rates, and sorbent material used to capture air samples.]

- Extraction method:

  [Include information regarding the retrievals of air samples.]

- Method validation (Including LOD and LOQ):

  [Include information and study report title of any independent laboratory validations. Also, describe the establishment of the limits of detection and limits of quantification for air samples.]

- Instrument performance:

  [Include information on calibration standards for the detection method.]

8. Quality Control for Air Sampling

Lab Recovery: [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 $\pm$ 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).]

Field blanks: [Describe any background levels of test substance measured before application(s) in the study.]

Field Recovery: [Most/All] field spike recoveries are within the acceptable range with overall recoveries between [x and y percent]. The exceptions include field spikes extracted during [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 field spike extractions (less than 90 percent or greater than 110 percent).]

Travel Recovery: [Describe the number of travel spikes prepared along with the fortification levels included. Describe the range of recoveries measured from the spiked samples.]

Breakthrough: [Describe any analysis or instances of test substance breakthrough in air sampling tubes, if applicable.]

9. Application Verification

Briefly describe the application verification methods used, *e.g.*, petri dish, saturation pads, spray tank analysis, *etc.*

Recoveries achieved on extraction and analysis of application monitors were in the range [xx to xy %]. Recovery achieved on analysis of field spiked samples was [xx %].

II. Results and Discussion

A. Empirical Flux Determination Method Description and Applicability

[Describe the empirical approach the registrant used to determine the flux rates as well as their appropriateness to the study design. Consider the below excerpts for various methods that have been developed for past reviews:]

<Indirect Method>

The indirect method, commonly referred to as the "back calculation" method, was the technique employed for estimating flux rates from fields treated for this field study given the available data. In the indirect method, air samples are collected at various locations outside the boundaries of a treated field. Meteorological conditions, including air temperature, wind speed, and wind direction, are also collected for the duration of the sampling event. The dimensions and orientation of the treated field, the location of the samplers, and the meteorological information are used in combination with the ISCST3 dispersion model (Version 02035) and a unit flux rate of 0.001 µg/m^{2}·s to estimate concentrations at the sampler locations. Since there is a linear relationship between flux and the concentration at a given location, the results from the ISC model runs are compared to those concentrations actually measured, and a regression is performed, using the modeled values along the x-axis and the measured values along the y-axis. If the linear regression does not result in a statistically significant relationship, the regression

may be rerun forcing the intercept through the origin, or the ratio of averages between the monitored to modeled concentrations may be computed, removing the spatial relationship of the concentrations. The indirect method flux back calculation procedure is described in detail in Johnson et al., 1999.

<Aerodynamic Method>

The aerodynamic method, also referred to as the "flux-gradient" method, was the technique employed for estimating flux rates from fields treated for this field study given the available data. In the aerodynamic method, a mast is erected in the middle of the treated field and concentration samples are typically collected at four or five different heights, ranging from 0.5 to 10 feet. Likewise, temperature and wind speed data are collected at a variety of heights. A log-linear regression is performed relating the natural logarithm of the sample height to the concentration, temperature, and wind speed. These relationships are then incorporated into an equation to estimate flux. The methods to estimate flux and related equations are presented in Majewski et al., 1990. The equation for estimating flux using the aerodynamic method is Thornthwaite-Holzman Equation, which is shown in the following expression:

Equation x1

$$P = \frac{k^2(\Delta\bar{c})(\Delta\bar{u})}{\phi_m\phi_p[\ln\left(\frac{z_2}{z_1}\right)]^2}$$

where P is the flux in units of $\mu g/m^2 \cdot s$, k is the von Karman's constant (dimensionless ~0.4), $\Delta\bar{c}$ is the vertical gradient pesticide residue concentration in air in units of $\mu g/m^3$ between heights z_{top} and z_{bottom} in units of meters, $\Delta\bar{u}$ is the vertical gradient wind speed in units of m/s between heights z_{top} and z_{bottom}, and ϕ_m and ϕ_p are the momentum and vapor stability correction terms respectively. Following the conditions expected in the neutrally stable internal boundary layer characterized by an absence of convective (buoyant) mixing but mechanical mixing due to wind shear and frictional drag, a log-linear regression is performed relating the natural logarithm of the sample height to the concentration, temperature, and wind speed. The adjusted values of the concentration, temperature, and wind speed from this regression is incorporated into Equation 2-1 to arrive at Equation 2-2 which is ultimately used to compute the flux.

Equation x2

$$Flux = \frac{-(0.42)^2(c_{ztop} - c_{zbottom})(u_{ztop} - u_{zbottom})}{\phi_m\phi_p \ln\left(\frac{z_{top}}{z_{bottom}}\right)^2}$$

where ϕ_m and ϕ_p are internal boundary layer (IBL)stability correction terms determined according to the following conditions based on the calculation of the Richardson number, R_i:

Equation x3

$$R_i = \frac{(9.8)(z_{top} - z_{bottom})(T_{ztop} - T_{zbottom})}{\left[\left(\frac{T_{ztop} + T_{zbottom}}{2}\right) + 273.16\right] + \left(u_{ztop} - u_{zbottom}\right)^2}$$

where T_{ztop} and $T_{zbottom}$ are the regressed temperatures at the top and bottom of the vertical profile in units of °C.

if R_i >0 (for Stagnant/Stable IBL)
$\phi_m = (1+16R_i)^{0.33}$ *and* $\phi_p = 0.885(1+34R_i)^{0.4}$

if R_i <0 (for Convective/Unstable IBL)
$\phi_m = (1-16R_i)^{-0.33}$ *and* $\phi_p = 0.885(1-22R_i)^{-0.4}$

The minimum fetch requirement that the fetch is 100 times the highest height of the air sampler for this method to be valid [was/was not] satisfied at all times [List the sampling periods and resulting fetches with exceptions]. The aerodynamic method used to estimate flux and related equations are presented in Majewski et al., 1990.

<Integrated Horizontal Flux Method>

The integrated horizontal flux method, also referred to as the "mass balance" method, was the technique employed for estimating flux rates from fields treated for this field study given the available data. In the integrated horizontal flux method, a mast is erected in the middle of the treated field and concentration samples are typically collected at four or five different heights, ranging from approximately 0.5 to 5 feet. Likewise, wind speed data are collected at a variety of heights. A log-linear regression is performed relating the natural logarithm of the sample height to the air concentration and wind speed following the log law relationships for the atmospheric boundary layer. These relationships are then incorporated into an equation to estimate flux. The methods to estimate flux and related equations are presented in Majewski et al., 1990. The equation for estimating flux using the integrated horizontal flux method is the following expression:

Equation x_1 $$P = \frac{1}{x}\int_{Z_0}^{Z_p} \bar{c}\bar{u}\,dz$$

where P is the volatile flux in units of μg/m^2·s, $\bar{c}$ is the average pesticide residue concentration in units of μg/m^3 at height Z in units of meters, $\bar{u}$ is the wind speed in units of m/s at height Z, x is the fetch of the air trajectory blowing across the field in units of meters, Z_0 is the aerodynamic surface roughness length in units of meters, Z_p is the height of the plume top in units of meters, and dz is the depth of an incremental layer in units of meters. Following trapezoidal integration, equation 3 is simplified as follows in equation 2-5 (Yates, 1996):

Equation x_2 $$P = \frac{1}{x}\sum_{Z_0}^{Z_p} (A * Ln(z) + B) * (C * Ln(z) + D)dz$$

where A is the slope of the wind speed regression line by ln(z), B is the intercept of the wind speed regression line by ln(z), C is the slope of the concentration regression by ln(z), D is the intercept of the concentration regression by ln(z), z is the height above ground level. Z_p can be determined from the following equation:

Equation x3 $$Z_P = \exp\left[\frac{(0.1 - D)}{C}\right]$$

The minimum fetch requirement of 20 meters for this method to be valid [was/was not] satisfied at all times [List the sampling periods and resulting fetches with exceptions]. [Describe the surface characteristics of the field and whether the maximum surface roughness length requirement of 0.1 meters for the method to be valid was satisfied. This condition is satisfied for studies conducted over bare soil or sod grass.]

B. Temporal Flux Profile

The flux determined from the registrant and reviewer for each sampling period after the application is provided in **Table 5**.

Table 5. Field volatilization flux rates of [test substance or analyte] obtained in study [Field or Plot, Application]

Sampling Period	Date/ Time	Sampling Duration (hours)	Flux Estimate			
			Reviewer	Registrant	Empirical Flux Determination Method	Notes
1	MM/DD/ YY [##:##] – [##:##]	[#]	[#]	[#]	[ID, AD, IHF, *etc*.]	[Include notes on missing data, or selection of reasoning used for Indirect Method.]
2	MM/DD/ YY [##:##] – [##:##]	[#]	[#]	[#]	[ID, AD, IHF, *etc*.]	[Include notes on missing data, or selection of reasoning used for Indirect Method.]
3	MM/DD/ YY [##:##] – [##:##]	[#]	[#]	[#]	[ID, AD, IHF, *etc*.]	[Include notes on missing data, or selection of reasoning used for Indirect Method.]
4	MM/DD/ YY [##:##] – [##:##]	[#]	[#]	[#]	[ID, AD, IHF, *etc*.]	[Include notes on missing data, or selection of reasoning used for Indirect Method.]
5	MM/DD/ YY [##:##] – [##:##]	[#]	[#]	[#]	[ID, AD, IHF, *etc*.]	[Include notes on missing data, or selection of reasoning used for Indirect Method.]
n	MM/DD/ YY [##:##] – [##:##]	[#]	[#]	[#]	[ID, AD, IHF, *etc*.]	[Include notes on missing data, or selection of reasoning used for Indirect Method.]

*****Methods legend: ID = Indirect method, AD = Aerodynamic Method, IHF = Integrated Horizontal Flux.

[Add on to tables as necessary for different locations, plots, or applications.]

[List specific maxima in volatile flux rates and possible factors contributing to these off-gasing events].

[Describe any abnormalities leading to potential uncertainties in the flux determinations such as missing data, low statistical significance between modeling and air sampling data in the indirect method, or low r-squared values in log-linear vertical profiles of temperature, wind speed, and concentration in any of the on-field methods (*e.g.*, aerodynamic or integrated horizontal flux methods).]

III. Study Deficiencies and Reviewer's Comments

[List any deficiencies with the study and any additional salient information. Specifically mention any aspects of the study that lead to uncertainties in the determination of flux rates.]

IV. References [List any references cited in the review.]

Johnson, B., Barry, T., and Wofford P. 1999. Workbook for Gaussian Modeling Analysis of Air Concentrations Measurements. State of California Environmental Protection Agency, Department of Pesticide Regulation. Sacramento, CA.

Majewski, M.S., Glotfelty, D.E., Kyaw Tha Paw U., Seiber, JN. 1990. A field comparison of several methods for measuring pesticide evaporation rates from soil. Environmental Science and Technology, 24:1490-1497.

Wilson, J.D., and Shum. W.K.N. 1992. A re-examination of the integrated horizontal flux method for estimating volatilisation from circular plots. Agriculture Forest Meteor. Vol 57:281-295.

Yates, S.R., F.F. Ernst, J. Gan, F. Gao, and Yates, M.V. 1996. Methyl Bromide Emissions from a Covered Field: II. Volatilization," Journal of Environmental Quality, 25: 192-202.

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 or refer to this table if it exists in a separate, associated document. Do not include in the table multiple versions of chemical names, structures, or 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. 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.]

Degradate Table.docx

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.]

[Example Excel files and spreadsheets follow below.]

1. Validation spreadsheet for studies following the Indirect Method:

Indirect Method.xlsx

2. Validation spreadsheet for studies following the Aerodynamic Method:

Aerodynamic Method.xlsx

3. Validation spreadsheet for studies following the Integrated Horizontal Flux Method:

IHF Method.xlsx

Attachment 3: Field Volatility Study Design and Plot Maps

[Insert map of study plot(s) from study report here.]

Attachment 4: Calculations and Index of Variables Used in Flux Determination Methods

Aerodynamic Method

Equation x_1

$$Flux = \frac{-(0.42)^2 (c_{ztop} - c_{zbottom})(u_{ztop} - u_{zbottom})}{\phi_m \phi_p \ln\left(\frac{z_{top}}{z_{bottom}}\right)^2}$$

Flux ($\mu g/m^2 s$): volatile flux of pesticide from release source surface

c_{ztop} ($\mu g/m^3$): concentration at the top sampler adjusted according to the regression of concentration vs. ln (height)

$c_{zbottom}$ ($\mu g/m^3$): concentration at the bottom sampler adjusted according to the regression of concentration vs. ln (height)

u_{ztop} (m/s): wind speed at the top sampler adjust according to the regression of wind speed vs. ln (height)

$u_{zbottom}$ (m/s): wind speed at the top sampler adjust according to the regression of wind speed vs. ln (height)

ϕ_m and ϕ_p (dimensionless): Internal Boundary Layer (IBL) stability correction terms determined according to the following conditions based on the calculation of the Richardson number, R_i:

Equation x_2

$$R_i = \frac{(9.8)(z_{top} - z_{bottom})(T_{ztop} - T_{zbottom})}{\left[\left(\frac{T_{ztop} + T_{zbottom}}{2}\right) + 273.16\right] + \left(u_{ztop} - u_{zbottom}\right)^2}$$

where:

T_{ztop}: Temperature at the top sampler adjusted according to the regression of temperature vs. ln (height)

$T_{zbottom}$: Temperature at the bottom sampler adjusted according to the regression of temperature vs. ln (height)

R_i (dimensionless): Richardson Number

if $R_i > 0$ (for Stagnant/Stable IBL)

$$\phi_m = (1 + 16R_i)^{0.33} \text{ and } \phi_p = 0.885(1 + 34R_i)^{0.4}$$

if $R_i < 0$ (for Convective/Unstable IBL)

$$\phi_m = (1 - 16R_i)^{-0.33} \text{ and } \phi_p = 0.885(1 - 22R_i)^{-0.4}$$

Integrated Horizontal Flux Method

Equation x_3

$$P = \frac{1}{x}\sum_{Z_0}^{Z_P} (A * Ln(z) + B) * (C * Ln(z) + D)dz$$

P ($\mu g/m^2 s$): volatile flux of pesticide from release source surface
z (m): height above ground level
A (s^{-1}): slope of the wind speed regression line by ln(z)
B (m/s): intercept of the wind speed regression line by ln(z)
C ($\mu g/m^4$): slope of the concentration regression by ln(z)
D ($\mu g/m^3$): intercept of the concentration regression by ln(z)
Z_0 (m): aerodynamic surface roughness length of release source surface
Z_p (m): volatile plume top height; calculated from the following equation:

Equation x4 $$Z_P = \exp\left[\frac{(0.1 - D)}{C}\right]$$

Section II. Field Volatility Study Review Considerations

This list of considerations is provided to help reviewers assess the acceptability of field volatility studies. This list is not a screen or a checklist and is not meant to be attached to the study reviews. Listed considerations carry unequal weight. Evaluate them using best professional judgment. Consider all information from the study and from similar studies to determine whether any deficiencies affect the study classification.

- The test sites were located in the United States or in ecoregions representative of domestic (USA) sites.
- A Typical End Use Product (TEP) was used, or an adequate explanation provided to justify the alternative chosen.
- Test substance was added to the soil at the highest recommended label rate for a single application.
- The test site (including soil type) was typical of actual use; soil was adequately characterized using the USDA classification system.
- The sites used for this study were clearly shown to have no previous use history involving this or closely related compounds or was clearly shown to contain no background residues or analytical interferences.
- Study was conducted under required labeled or typical use conditions (*e.g.*, time of year, time of day, typical agricultural practices).
- The study design included an untreated replicate subplot.
- Irrigation: The study design includes irrigation according to the label requirements.
- An adequate number of sites were used (generally 4-6) in different areas representative of the intended usage. Fewer sites are required for limited use patterns.
- Soil texture was adequately characterized using the USDA classification system.
- Soil bulk density (g/cm^3), soil temperature (°C), soil moisture (percent of field capacity), and organic carbon content (percent) were reported at numerous depths.
- Soil taxonomy was adequately characterized using the USDA or, for foreign soils, the WRB classification system (Citation: USEPA, 2011).
- Air sampling intervals were adequate.
- Volatility was reported in units of ($\mu g/m^2 \cdot s$).
- The concentration of the AI and degradates of concern were measured in air.
- Concentrations in air were reported in units of ($\mu g/m^3$).
- Appropriate analytical methods were provided.
- Detection and quantitation limits were reported.

- Field, travel, and laboratory spiked samples were deployed and related data were provided. An adequate number of spiked samples at multiple fortification levels were sampled consistent with the sampling periods.
- A storage stability study was conducted using either spiked field and spiked laboratory samples to determine the stability of samples under typical lab storage conditions. This study was conducted for at least as long as the longest duration of field sample storage.
- Raw measured air concentration data were provided, including vacuum pump flow rates (in units of L/min), sampling intervals, mass detections in tubes (in units of μg), and representative chromatographs.
- Air monitoring started at the beginning of the application period.
- On-site meteorological data were measured.
- All meteorological data were sampled at a frequency of at least 1 minute.
- Wind speed measurements were reported in units of m/s. Temperature measurements were reported in units of °C.
- A reasonable attempt was made to identify the parent and all major degradates identified from laboratory studies.
- An independently validated environmental chemistry method(s) was/were submitted to evaluate the precision and accuracy of the analytical method(s) used in the field study.
- A plot layout showing all fields treated as well as the layout of air samplers and meteorological instrumentation with all appropriate distances was provided.
- For the off-field flux monitoring approach, the appropriate dispersion model was used to back-calculate the flux (*e.g.*, ISCST3 for bare fields and AERMOD for cropped fields).
- For on-field flux monitoring approaches, appropriate minimum fetches and maximum surface roughness length requirements for the appropriate empirical flux determination method to be valid were satisfied at all times throughout the study.

References

U.S. Environmental Protection Agency (USEPA). 2011. Guidance for Determining the Acceptability of Environmental Fate Studies Conducted with Foreign Soils. May 20, 2011. (http://www.epa.gov/pesticides/science/efed/policy_guidance/team_authors/environmental_fate_tech_team/soils_foreign.htm, accessed Dec. 12, 2012)

Fish and Oyster BCF Study Review Guide

This guide is meant to aid in the review of fish and oyster bioconcentration studies submitted under the Office of Chemical Safety and Pollution Prevention (OCSPP) guidelines 850.1710 (Oyster BCF) and 850.1730 (Fish BCF)[1]. The BCF study review format is loosely based on the format of the Organisation for Economic Co-operation and Development (OECD) Tier II Summaries (T2S)[2], although an OECD T2S format specific to BCF studies is not available currently. In developing study reviews, reviewers should strive to write concisely and to minimize alterations to the T2S format.

Sections I and II of this document provide details regarding data, formatting, and issues to consider in developing OCSPP environmental fate study reviews. Although this guide is not intended to strictly prescribe where and how to present the data, an example study review template is provided in Section I for guidance. In general, reviewers should follow the example template, especially for unilateral reviews (*i.e.*, reviews conducted without the participation of other agencies). However, reviewers may modify the template as needed or disregard it in the case of multilateral reviews (*i.e.*, reviews with other agencies participating) in which an alternative format is agreed upon by the participating agencies. In Section II of this document, a list of review considerations is provided to help reviewers focus on critical study issues and to identify any common major or minor deficiencies.

References

Organisation for Economic Cooperation and Development (OECD). 1996, *Test No. 305: Bioconcentration: Flow-through Fish Test*, OECD Guidelines for the Testing of Chemicals, Section 3, OECD Publishing. Dated June 14, 1996. (DOI: 10.1787/9789264070462-en, accessed Nov. 30, 2012)

OECD. 2012. Draft Technical Guidance: Bioaccumulation in Fish: Aqueous and Dietary Exposure. OECD Guidelines for the Testing of Chemicals, Section 3: Degradation and Accumulation, Adopted by WNT, OECD Publishing (ENV/JM/TG(2012)31 dated March 13, 2012). (http://www.oecd.org/chemicalsafety/testingofchemicals/50309198.pdf, accessed Nov. 30, 2012)

OECD. 2012. Bioaccumulation in Fish: Aqueous and Dietary Exposure. OECD Guidelines for the Testing of Chemicals, Section 3: Degradation and Accumulation, Adopted by WNT, OECD Publishing. Dated October 2, 2012. (DOI : 10.1787/9789264185296-en, accessed Nov. 30, 2012)

[1] OCSPP guidelines 850.1710 and 850.1730 are not final currently.

[2] A Tier II Summary is an OECD format for study reviews prepared by industry. See OECD Guidance Documents for Pesticide Registration at http://www.oecd.org/chemicalsafety/agriculturalpesticidesandbiocides/oecdguidancedocumentsforpesticideregistration.htm (Accessed Dec. 12, 2012).

U.S. Environmental Protection Agency (USEPA). 1996. Fish BCF. OCSPP 850.1730 Harmonized Test Guidelines (formerly OPPTS 850.1730). Dated April 1996. (http://www.epa.gov/ocspp/pubs/frs/home/draftguidelines.htm, accessed Nov. 30, 2012)

USEPA. 1996. Oyster BCF. OCSPP 850.1710 Harmonized Test Guidelines (formerly OPPTS 850.1710). Dated April 1996. (http://www.epa.gov/ocspp/pubs/frs/home/draftguidelines.htm, accessed Nov. 30, 2012)

USEPA. 2009. User's Guide and Technical Documentation, KABAM version 1.0, (K_{OW} (based) Aquatic BioAccumulation Model). Environmental Fate and Effects Division, Office of Pesticides Program. Dated April 7, 2009. (http://www.epa.gov/oppefed1/models/water/kabam/kabam_user_guide.html, accessed Nov. 30, 2012)

Section I. Example Fish and Oyster BCF Study Review Template

[Fish or Oyster] Bioconcentration Factor (BCF) of [Test Compound]

Report:	[Provide full citation. Provide the MRID (first) if the review is unilateral.]
Document No.:	[MRID ######## (for the U.S.)] [PMRA Study No. ####### (for Canada)]
Guideline:	OCSPP [850.1710 for oysters or 850.1730 for fish in the U.S.]; DACO [9.4.8 for oysters or 9.5.6 for fish in Canada] [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 850.1710' ['850.1730']. 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 a 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:	[######]

Reviewer:	[Provide final reviewer(s)'s name]	**Signature:**
	[Title]	**Date:** [Type date of signature.]

Executive Summary

The bioconcentration and depuration of [location(s) of radiolabel(s)-^{14}C]-[test compound] in [common name of test species] was investigated in a [type of test system, *e.g.*, continuous flow-through or renewal] system. The [fish or oysters] were continuously exposed at nominal low and high dose concentrations of [##] µg/L and [##] µg/L, respectively, for [##] days at [##]°C. Concentrations in [fish or oysters], [reached or did not reach] a plateau after [##] days of exposure. The [fish or oysters] were then transferred to [flowing or renewed] untreated water after [##] days of study initiation to depurate for [##] days. [##] treated replicate tanks, [##] control tanks, and [##] solvent control tanks per concentration were tested.

[If bioconcentration factors (BCFs) were corrected for TOC in the water, then state it.] BCFs appeared to be [dependent or independent] of the water concentration. The [test compound] was a maximum of [##]% of the total residue recovered (TRR) at day [##] of exposure in the [fish or oyster] tissue for the high [or low] dose exposure samples. [No or [##]] transformation products were identified in the water and subsequently in the [fish or oyster] tissue (**Table 11**). Metabolism of [test compound] [occurred or did not occur] in the [fish or oyster] tissue as shown by the presence of [no or [##]] tissue metabolites in the [fish or oyster] tissue that were not

present in the water (**Table 11**). A synopsis of the study results is provided in **Table 1** and **Table 2**.

Table 1. [Test substance] [Fish/Oyster] Kinetic Parameters [report only the ones required]

Type of exposure	Continuous flow-through or renewal	
Time to steady state	[##] days	
Uptake rate constant k_1	[##]±[C.I.] $L \cdot Kg^{-1} \cdot day^{-1}$	
Depuration rate constant k_2	[##]±[C.I.] $days^{-1}$	
Growth rate constant k_G	[##]±[C.I.] $days^{-1}$ [usually required only for the fish BCF studies conducted for extended periods of time, *e.g.*, above 28 days]	
Metabolism rate constant k_M	[##]±[C.I.] $days^{-1}$ [usually required only for the fish BCF studies where there is considerable tissue metabolism]	
Fecal egestion rate constant k_E	0 $days^{-1}$ (considered negligible in BCF studies)	
$k_T = k_2 + k_M + k_G + k_E$	[##]±[C.I.] $days^{-1}$	
Depuration half-life	Low dose: [##]±[C.I.] days	High dose: [##]±[C.I.] days
Amount depurated	[##]% TRR after [##] days for the [low or high] dose samples	

k_T is the total elimination rate constant. Data obtained from pages [##] of the study report. Rate constants were calculated using the equations in Attachment I, **Table I-1**.

Table 2. [Test substance] [Fish/Oyster] Bioconcentration Factors [report the ones required]

Concentration:	**[##] µg a.i./L**			**[##] µg a.i./L**		
Tissue:	**Edible**	**Non-edible**	**Whole**	**Edible**	**Non-edible**	**Whole**
BCF_{SS} ($L \cdot Kg^{-1}$)*	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]
BCF_{K} ($L \cdot Kg^{-1}$)*	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]
BCF_{KG} ($L \cdot Kg^{-1}$)*	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]	[##]±[C.I.]
L (%)	[##]	[##]	[##]	[##]	[##]	[##]
$BCF_{SS, L}$ ($L \cdot Kg^{-1}$)*	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]	[##]±[S.D.]
$BCF_{K, L}$ ($L \cdot Kg^{-1}$)*	[##]	[##]	[##]	[##]	[##]	[##]
$BCF_{KG, L}$ ($L \cdot Kg^{-1}$)*	[##]	[##]	[##]	[##]	[##]	[##]

***** Units for BCFs: L/Kg wet weight tissue; units for lipid normalized BCFs are L/Kg lipid.
Data were obtained from pages [##] of the study report. BCF_{SS}, BCF_K and BCF_{KG} are the steady state, kinetic and growth corrected kinetic BCF; L is the lipid content which was determined at [##] days of exposure. BCFs were calculated using the equations in Attachment I, **Table I-1**.

[Notes to the reviewer: For the BCFs and kinetic constants, provide the confidence limits or standard deviations, where available, in **Table 1** and **Table 2**. BCF values are calculated from the concentration of test substance (instead of the total radioactivity), unless evidence is provided that the test material does not degrade. For further guidance on the correction for TOC in the dilution water, which is typically required when the log K_{OW} >4 and TOC is high, see the KABAM manual (**Equation *A*2** of Appendix A, which is from Arnot and Gobas, 2004) [3]. The oyster BCF guideline does not recommend calculation of k_G, BCF_{KG} or $BCF_{KG\,L}$. For fish, BCF_{KG} is needed only for extended studies. BCF_L and BCF_{KG} may or may not be available for individual tissues in the fish BCF study. For OCSPP BCF studies, k_E (rate constant for fecal egestion) is not usually measured and is assumed to be negligible. Be careful throughout the review to distinguish transformation products in the organisms from those in the water.]

[3] Arnot, J.A. and F.A.P.C. Gobas. 2004. A food web bioaccumulation model for organic chemicals in aquatic ecosystems. Environmental Toxicology and Chemistry, 23 (10): 2343-2355.

I. Study Design

A. Preliminary Tests

[Describe any preliminary tests performed such as a range finding, solubility or toxicity test.]

B. Materials

Provide a small image of the test compound's structure and any radiolabel on the right margin

Table 3. Materials

Test material	[Type of label]-radiolabeled [test compound]. Radiolabel position(s) [was/ was not/ were/were not] appropriate for this study.
Specific radioactivity	[##] MBq/mg
Radiochemical purity	[##]% [HPLC or specify method of determining purity]
Chemical purity	[##]% [HPLC, GC/MS or specify method of determining purity]
Batch number or ID	[xxxxxx]
Solubility in water	[##] mg/L [If pH-dependent, list available value at study pH; also list the solubility in saltwater if available and relevant (*e.g.*, for an oyster BCF study or for a fish BCF study conducted with a saltwater species).]
Hydrolysis half-life at pH 7	[##] days [provide other hydrolysis half-lives and/or aqueous photolysis half-life, if relevant]
pK_a	[##] [provide when appropriate]
Other	[Provide a brief description of any other issues that might have been encountered in the study or any other physicochemical or environmental fate parameters that may have affected the results. Describe detected impurities in the water.]

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

Table 4. Water Solutions

Water	[Brief description of the dilution water and its source, *e.g.*, filtered ([##] µm) well water, dechlorinated tap water, natural seawater, from [source], *etc.*]	
Temperature	Mean [##]±[##]°C; range [##]-[##]°C [report frequency of measurements]	
Flow rate	[##] mL per [##] hr or [##] L/hr [report frequency of measurements]	
Dissolved oxygen concentration	[##] to [##] mg/L; dissolved oxygen was maintained at ≥[##]% saturation (method) [report frequency of measurements]	
pH	[##] to [##] (method) [report frequency of measurements]	
Total organic carbon	[##] to [##] mg carbon/L [report frequency of measurements]	
Dissolved organic carbon	[##] to [##] mg carbon/L [report frequency of measurements]	
Particulate matter	[##] to [##] mg/L [report frequency of measurements]	
Hardness	[##] to [##] mg/L as $CaCO_3$ [report frequency of measurements]	
Alkalinity	[##] to [##] mEq/L [report frequency of measurements]	
Salinity	[##] to [##] ppt [report frequency of measurements; report salinity only for tests performed with estuarine/marine fish or with oysters]	
Test concentrations	Nominal low dose: [##] mg/L	Nominal high dose: [##] mg/L
Range	Low dose: [##]-[##] mg/L	High dose: [##]-[##] mg/L
Stock solution	[Indicate how the stock solution was prepared.]	
Water solution	[Indicate how the water was prepared.]	

Vehicle/Concentration	[name of vehicle used] at [##] mL/L
Surfactants	A surfactant or dispersant [was or was not] used in the preparation of a stock or water solution. [Identify the surfactant if used.]
Loading rates	[##], [##] and [##] g fish/L/day [or [##], [##] and [##] oysters/L/hour], for the low dose samples, high dose samples, and control, respectively. The loading rate [was or was not] compliant with the [Fish or Oyster] BCF guideline requirement.
Other	[Describe any other issues that might have been encountered in the study.]

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

Table 5. Testing System

Test type	[For example, continuous flow-through or renewal], [##] volume additions per day, [[##] flow rate for flow-through test.] The type of test system [was or was not] compliant with the [Fish or Oyster] BCF guideline requirements.
Test chambers	[Provide a description of the aquaria, volume, *e.g.*, [##]-L; material, *e.g.*, glass or stainless steel; and shape, *e.g.*, rectangular chambers. Indicate the dimensions of each aquarium.] All test vessels and compartments [had or did not have] the same dimensions and water volumes.
Number of aquaria	[##] treated aquaria, [##] control, and [##] solvent (vehicle) control.
Randomization	Treatments [were or were not] randomly assigned to individual test vessel locations and individual test organisms [were or were not] randomly assigned to test vessels.
Aeration	Aeration [was or was not] used [If aeration was used, describe in brief; note that aeration is not recommended.]
Light source	[xxxx]
Intensity of light	[##] to [##] [unit (*e.g.*, ft-c or lux)]
Photoperiod	[##] hours of light:[##] hours dark
Other	[Describe any other issues that might have been encountered in the study.]

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

Table 6. Test Organisms

Test organism	[Provide the common name of the species.]
Species	[Provide the *Scientific name* of the species.]
Weight [or Valve height]	Mean [##]±[##] g; range [##] to [##] g [For oyster BCF: Substitute for valve height range in mm.] [For fish BCF: the registrant may have provided the length in lieu of the weight (note that the guidance recommends measuring both). If this is the case, report the length and the registrant's justification for not reporting the weight of the test organisms.]
Size	[For fish BCF: The smallest fish [was or was not] smaller than ⅔ the weight of the largest. For oyster BCF: The organisms [were or were not] within the recommended range of 30-50 mm in valve height and the standard deviation was less than 20% of the mean.]
Age/Life Stage	[xxxx]/[xxxx] [For fish BCF: It is recommended to use fish of the same year-class; if the fish were not juveniles, provide a justification for using older fish]
Source	[xxxx]][It is recommended that all fish or oysters proceed from the same source.]
EC_{10}, EC_{50}, IC_{10}, IC_{50},or LC_{50}	[##] µg/L [Provide the appropriate values from guideline studies and citation (*e.g.*, MRID [########]) or from preliminary experiments conducted and reported in this study.]

Acclimation period	[##] days [A holding period of at least 14 days for fish BCF, and 12 days for oyster BCF, is recommended]
Diet	[For example, for fish BCF: Test organisms were fed approximately [##]% of the fish body weight per day. Uneaten food and feces was siphoned [##] minutes after being fed. Or for oyster BCF: Feeding regime was continuous via delivery of dilution water (*e.g.*, natural unsterilized and unfiltered seawater, or supplemented artificial seawater).]
Treatment for disease	[No or [xxxx]] treatment for disease was conducted through the acclimation or testing periods.
Mortality for the treated samples	[##] ([##]%) for the treated samples at the low dose concentration; [##] ([##]%) for the treated samples at the high dose concentration.
Mortality for the controls	[##] ([##]%) for the vehicle (solvent) control; [##] ([##]%) for the control.
Frequency of observations	Mortality observations were conducted [frequency] for treated and control samples. [Mortality should be recorded at least daily]
Other observations	[Report any other observations such as spawning, any evidence of adverse effects, and lack of feeding, such that chemical uptake and/or depuration were likely impacted.]
Other	[Describe any other issues that might have been encountered in the study.]

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

C. Sampling and Analysis

1. Water

Table 7. Water Sampling and Analysis

Number of replicates sampled	[Duplicate for fish BCF or triplicate for oyster BCF] treated and control water samples were taken from each tank at each sampling interval. [See also **Table 5** (Testing System) for number of aquaria.]
Sampling intervals	At days -[##] and -[##], after 0, [##], [##], [##], [##], and [##] days of exposure, and after 0, [##], [##], [##], and [##] days of depuration. Water samples [were or were not] collected before feeding and at the same time that fish were collected. Initial water samples [were/were not] collected prior to the addition of fish/oyster to the test chamber.
Solvent (vehicle) sampling	Solvent (vehicle) control samples were taken at 0, [##], and [##] days.
Sample volume	[##] mL
Sample collection method	Samples were collected from each tank using [test equipment, *e.g.*, a volumetric pipette].
Analysis	Aliquots ([##] mL) were analyzed [*e.g.*, for total radioactivity using LSC, or appropriate analytical method].
LOD / LOQ for LSC in water	[##] µg/L / [##] µg/L [report results for low dose and high dose samples if they are different], equivalent to [##]% / [##]% of the TRR.
Additional analysis	[On days [##] and [##] of exposure, additional water samples ([##] samples, volume [##] L) were collected from each test aquarium and tested for [test compound] and [transformation products]. The analysis was performed [provide a synopsis of the analytical method].]
LOD / LOQ for parent compound in water	[##]µg/L/[##] µg/L (report results for low dose and high dose samples if they are different). Equivalent to [##]% / [##]% of the TRR.

LOD / LOQ for transformation products	[##]µg/L/[##] µg/L (report results for low dose and high dose samples if they are different). Equivalent to [##]% / [##]% of the TRR.
Raw data	Raw measured data and representative chromatographs [were or were not] provided.
Other	[Describe any other issues that might have been encountered in the study.]

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

2. [Fish or Oyster] Tissue

Table 8. [Fish or Oyster] Sampling and Tissue Analysis

Number of [fish or oysters] sampled at each interval	[##] for treated samples, [##] for solvent controls, and [##] for the control tank. [Note: usually 4 replicates are taken, for each test interval, except for the last exposure interval, when 6 replicates are taken. Additional replicates may be taken for lipid determination if it is not possible to measure it from the same fish or oysters tested.]
Pooling	Samples [were or were not] pooled. [If samples were pooled, describe.]
Sampling intervals	At 0, [##], [##], [##], [##], and [##] days of exposure, and after 0, [##], [##], [##], and [##] days of depuration
Sample handling	The [fish or oysters] were sacrificed, [for fish BCF weighted and measured or for oyster BCF valve height was measured], [for fish BCF add: and separated into fillet (edible tissue) and viscera (non-edible tissue)].
Methods of extraction and cleanup	Aliquots (*ca*. [##]-[##] g) were [methods, *e.g.*, dried overnight, extracted with [solvent], samples [cleaned up] using [method]] and [additional methods, [*e.g.*, combusted] prior to [method, *e.g.*, LSC] analysis. [Combustion or method] efficiency was [##]%.]
Analytical methods	Samples of days [##], [##] and [##] were further analyzed for parent compound and transformation products [or metabolites] using [method, *e.g.*, HPLC, GC/MS] analysis. [HPLC] extraction efficiency was [##]%. A reasonable attempt [was or was not] made to extract the test compound and its transformation products and/or metabolites from the tissue tested. [If a reasonable attempt was not made to extract the samples, refer to the Study Deficiencies (**Section IV**) for further details.]
LOD / LOQ for LSC analysis in [fish/oyster] tissue	[##]% of the TRR / [##]% of the TRR (report results for low dose and high dose samples if they are different).
LOD / LOQ for parent compound in [fish/ oyster] tissue	[##]µg/Kg or [##]% of the TRR /[##] µg/Kg or equivalent to [##]% of the TRR (report results for low dose and high dose samples if they are different).
LOD / LOQ for transformation products in [fish/oyster] tissue	[##]µg/Kg or [##]% of the TRR /[##] µg/Kg or equivalent to [##]% of the TRR (report results for low dose and high dose samples if they are different).
Raw data	Raw measured data and representative chromatographs [were or were not] provided.
Other	[Describe any other issues that might have been encountered in the study.]

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

3. Lipid Determination

[Briefly describe the method used for lipid determination. Provide citations for methods where applicable in **Section V**.]

4. Supplementary Studies

[Briefly describe any other supplementary studies and their results if any.]

II. Analytical Results

A. Findings

Based on a log K_{OW} = [##] for [test substance] at pH [##], bioconcentration was expected to plateau after [##] days of exposure. Steady state [was or was not] operationally achieved after [##] days of exposure, since after [##] consecutive measurements [minimum 3 measurements], separated by [##] days [usually 2-7 days], a plot of [test substance] concentration in whole fish on a wet weight basis (C_f), against time [became or did not become] nearly parallel to the time axis. They were within ±20% of each other [if after 3 measurements the concentration is still increasing, an additional intervals are recommended to document steady state; furthermore, when samples are pooled, 4 consecutive measurements are the minimum required to document steady state] (**Table 9** and **Figure 1**).

Table 9. [Test Substance] [Fish or Oyster] Residue Analysis (Exposure Phase)

Days	Exposure					
	0	**[##]**	**[##]**	**[##]**	**[##]**	**[##]**
Water TWA_C (µg/L)	[##]	[##]	[##]	[##]	[##]	[##]
C_f (µg/kg wet wt)	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]
%TRR	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]
BCF (L·Kg wet wt^{-1})	[##]	[##]	[##]	[##]	[##]	[##]

Data obtained from pages [##] of the study report. TWA_C=time weighted average concentration; C_f [or C_o] is the [test substance concentration] in whole fish [or oyster] on a wet weight basis. TRR=total residue recovered. TWA_C is calculated according to equation in **Table I-1** of Attachment I.

[If bioconcentration factors (BCFs) were corrected for total organic carbon (TOC) in the water, then state it. If the water analysis is based on LSC as opposed to individual residue analysis due to test substance's persistence, provide results of analyses for transformation products for representative samples, and proof that the levels of parent compound were reasonably related to the total radioactive residues in the water (*e.g.*, [TLC, HPLC, GC/MS] analysis showed that [^{14}C]-[test substance] accounted for ≥[##]% of the TRR and no additional individual residue analyses were conducted in the water samples.) [Provide a description of any transformation products or impurities in the water.] A summary of kinetic parameters and bioconcentration factors is provided in **Table 1** and **Table 2**, respectively, of the Executive Summary.

The period of depuration and sampling intervals [were or were not] adequate to determine a depuration rate constant and a depuration half-life for the parent compound. After [##] days of depuration, [##]% of the TRR and [##]% of the parent compound remained in the fish tissue (**Table 10** and **Figure 1**).

Table 10. [Test Substance] [Fish or Oyster] Residue Analysis (Depuration Phase)

Days of depuration	Depuration				
	0	[##]	[##]	[##]	[##]
C_f (µg/kg wet wt)	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]
%TRR	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]	[##]±[##]

Data obtained from pages [##] of the study report. [C_f or C_o] is the [test substance] concentration in [whole fish or oyster] on a wet weight basis. TRR=total residue recovered.

A plot of C_f vs. time is shown in **Figure 1** [example plot]:

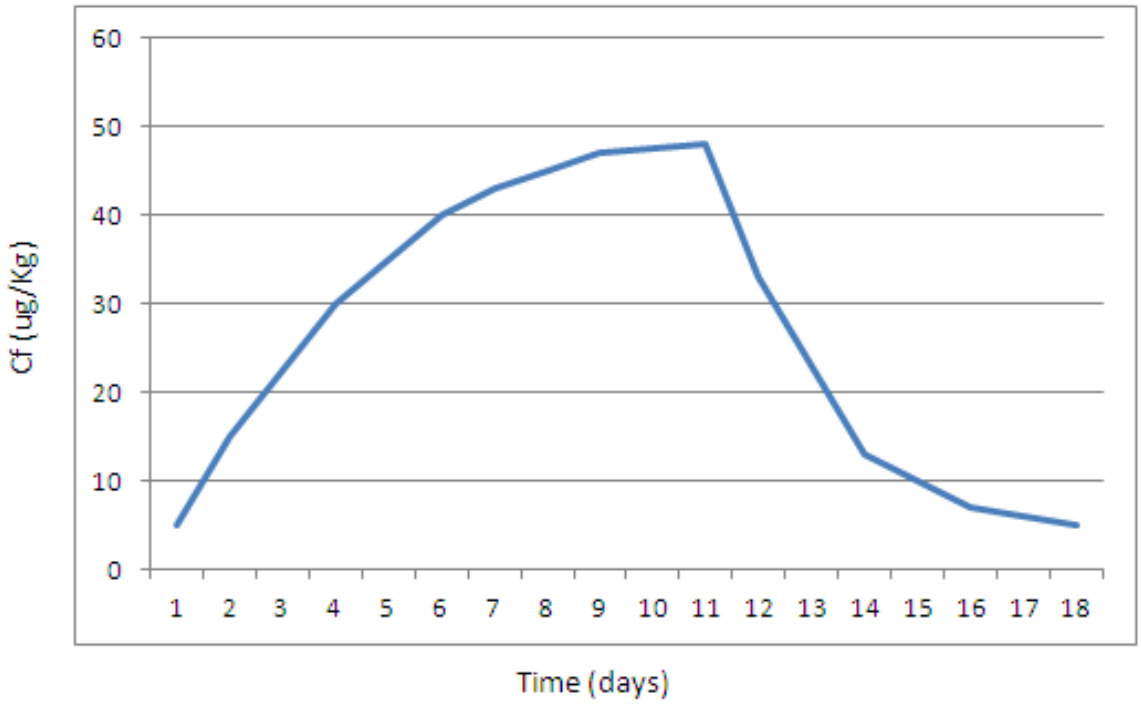


Figure 1. Concentration of [test substance] in whole fish (C_f, µg/Kg) vs. time (days)

B. Bioconcentration of Transformation Products and/or Metabolites

[If the BCF_{SS} is ≥500 and transformation products or metabolites exceed 10% TRR, then they are identified. Furthermore, any compound of toxicological concern is also measured, regardless of the percent of the TRR. Use this section and the sample **Table 11** to report this data.]

Table 11. Concentration in [Fish or Oyster] of [Test Substance] and Transformation Products

Tissue	[Edible, Non-edible or Whole Fish or Oyster Tissue, as applicable]				
Name	Max. Conc. (µg/g)	Interval (days)	Maximum % of TRR	Max. Conc. After Depuration (µg/g)**	% of TRR After Depuration**
[Test substance]	[##]	[##]	[##]	[##]	[##]
[Compound #1]	[##]	[##]	[##]	[##]	[##]
[Compound #2]	[##]	[##]	[##]	[##]	[##]
[Compound #3]	[##]	[##]	[##]	[##]	[##]
NERs	N/A	[##]	[##]	N/A	[##]

Data obtained from pages [##] of the study report. TRR = total residues recovered; NERs = non-extracted residues. ******Value was measured after [##] days of depuration.

Maximum unextracted (or non-extracted) residues (NERs) were [##]% TRR. [If unextracted residues were ≥10% TRR, then indicate whether the extraction effort was exhaustive. Further discussion may be placed in the **Study Deficiencies** section below.]

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

[Self explanatory]

Attachment 1: Equations

Table I-1. Equations

Box I-1A. Calculation of k_1 and k_2

$$\ln C_{f\,dep} = \ln C_{f0} - (k_2)\,(t_{dep})$$

k_2 is derived from the slope of $\ln C_{f\,dep}$ vs. t_{dep};

[C is the concentration of the test substance in ["*f*" fish ("*o*" oyster), or "*w*" water]; t is the time for "*up*" uptake or "*dep*" depuration. C_{f0} is the concentration in fish at the time 0 (zero) when depuration starts. k_1 and k_2 are the uptake and depuration rate constants, respectively.]

Box I-1B. Calculation of k_G

Calculation of the growth rate constant, k_G (required for fish BCF studies conducted for extended periods of time, *e.g.*, exposure and depuration above 28 days): Plot the natural logarithm of each individual fish weight (*e.g.*, kg) against time (days) or the natural logarithm of each fish length (*e.g.*, mm) against time (day) when the weight is not available. The slope of the line is k_G, the growth rate constant ($days^{-1}$). Use all individual fish available in this calculation (control, and two test concentrations). See OCSPP 850.1730 for further guidance.

Box I-1C. Calculation of k_M

Calculation of the metabolism rate constant, k_M (required for fish BCF studies when there is considerable metabolism): The metabolism rate constant (k_M) may be calculated using one of the methods described in Appendix H of the KABAM user's manual (Methods for Estimating Metabolism Rate Constant (k_M))[4]. For further guidance, see the manual and Arnot *et al.*, 2008, along with the supplementary materials for the article.[5]

Box I-1D. Calculation of BCF_{SS}, BCF_K and BCF_{KG}

BCF_{SS} = Steady state bioconcentration factor = Mean BCF over the time period when steady state is achieved or $BCF_{SS} = C_{f,SS}/C_w$ (for derivation of C_w, the time weighted average concentration, see the equations in the **Box I-1F**)

BCF_K = Kinetic bioconcentration factor = k_1/k_2

BCF_{KG} = Growth corrected bioconcentration factor = $k_1/(k_2 - k_G)$ (assuming that $k_T = k_2 + k_G$ and k_M and k_E, the fecal egestion rate constant, are negligible)

[4] http://www.epa.gov/oppefed1/models/water/kabam/kabam_user_guide.html

[5] Arnot, J.A., D. Mackay, M. Bonnell. 2008. Estimating metabolic biotransformation rates in fish from laboratory data. *Environ. Toxicol. Chem.* 27 (2), 2008, pp. 341–351.

Table I-1. Equations

Box I-1E. Lipid Normalization

L is the lipid content in fish [or oyster] tissue (unitless or percent).

$C_{f, L}$ = lipid normalized concentration in fish [or "*o*" oyster if appropriate] = C_f/L

BCF_L = lipid normalized BCF = $C_{f, L}/C_w$ = BCF/L
$BCF_{SS, L}$ = lipid normalized steady state BCF = BCF_{SS}/L
$BCF_{K, L}$ = lipid normalized kinetic BCF = BCF_K/L
$BCF_{KG, L}$ = lipid normalized growth corrected kinetic BCF = BCF_{KG}/L

Box I-1F. Calculation of Time Weighted Average Concentration

TWA_C = time weighted average water concentration, calculated after [##] days of exposure:

Where the carried weight w_i is the period of time ($t_i - t_{i-1}$) or the number of hours or days at the concentration x_i; and x_i is the average concentration [$(C_j + C_{j-1})/2$].

Attachment 2: 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. Multiple versions of chemical names and SMILES strings are not 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 analytes.

For multilateral reviews, chemical names, SMILES strings, structures, and CAS numbers are captured elsewhere in the Monograph.[6] 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.]

Degradate Table.docx

[6] 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.

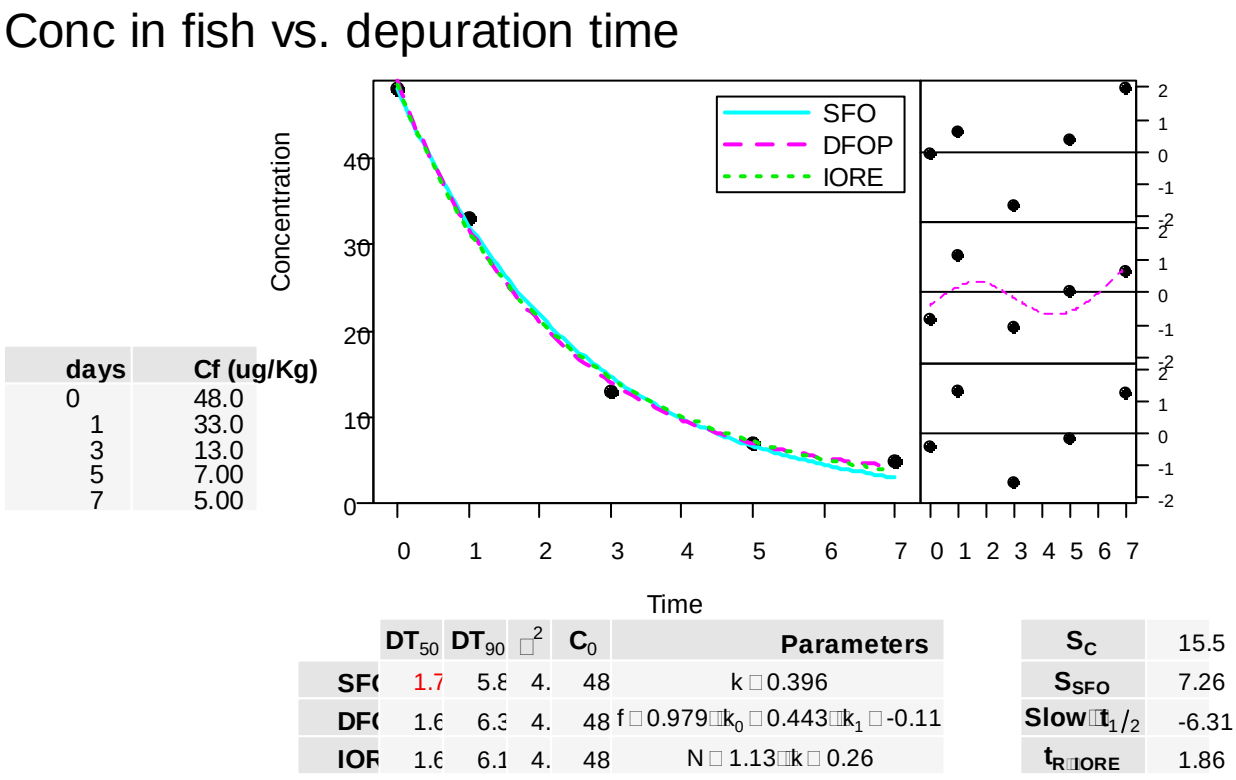
Attachment 3: Statistics Spreadsheets and Graphs

[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.]

[If the PestDF Tool is used, include the output files (note that PestDF is intended to describe degradation, while the process described in this study is depuration, which is assumed to follow single first order (SFO) kinetics). Output images can be pasted to the study review (as done in the example below), to the Excel spreadsheet, or attached as individual files with appropriate file names and extensions (*e.g.*, JPEG).]

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

[Example plot for the same data set shown in **Figure 1** above (choose SFO kinetics, $t_{1/2}$=1.75 days):]



days	Cf (ug/Kg)
0	48.0
1	33.0
3	13.0
5	7.00
7	5.00

	DT_{50}	DT_{90}	χ^2	C_0	Parameters
SFO	1.7	5.8	4.	48	k 0.396
DFO	1.6	6.3	4.	48	f 0.979 k_0 0.443 k_1 -0.11
IOR	1.6	6.1	4.	48	N 1.13 k 0.26

S_C	15.5
S_{SFO}	7.26
Slow $t_{1/2}$	-6.31
$t_{R\ IORE}$	1.86

Section II. Fish and Oyster Bioconcentration Study Review Considerations

This list of considerations is provided to help reviewers assess the acceptability of fish or oyster BCF studies. This list is not a screen or a checklist and is not meant to be attached to the study reviews. However, well written study reviews contain enough information to determine the status of each of these considerations. Listed considerations carry unequal weight. Evaluate them using best professional judgment. Consider all information from the study and from other relevant environmental fate studies (*e.g.*, if appropriate, hydrolysis, aqueous photolysis) to determine whether any deficiencies affect the study classification. Note that this DER Guide is based on OCSPP guidelines 850.1710 and 850.1730, which are not final currently.

Test Material

- The test material was Technical Grade (TGAI) or a Pure Active Ingredient Radiolabeled (PAIRA) compound.
- The radiopurity and specific activity of the test compound were provided (if radiolabeled).
- Radiolabel positioning was appropriate. If the compound contains ring structures, experiments were conducted separately for each respectively labelled ring structure, and test compounds were not radiolabeled on more than one ring structure in each experiment. (Typically required for tests performed with radiolabeled compounds for which degradation or metabolism is expected or occurred, and the BCF ≥500 L/Kg, for their identification.)

Test Organisms

- The appropriate species was used (*e.g.*, for **fish BCF** studies: bluegill sunfish or rainbow trout, or another freshwater or marine test species as specified in the guideline; for **oyster BCF** studies: Eastern oyster).
- Organisms were of uniform size (weight for fish BCF or valve height for oyster BCF). **Fish BCF:** smallest fish is not <⅔ weight of largest. **Oyster BCF:** the organisms were 30 to 50 mm in valve height, similar in age and/or size, and the standard deviation of valve height was less than 20 percent of the mean.
- For **fish BCF** studies, juvenile fish were preferred or an adequate explanation or justification as to why older fish were selected was provided.
- The number of organisms per concentration was sufficient to provide a minimum of 4 fish or 4 oysters per treatment on each sampling interval.
- **Fish BCF:** less than 10% of the organisms in either the dilution water or solvent control treatment showed mortality or sublethal effects. Death or other adverse effects were less than 5% per month or did not exceed 30% in all organisms for a test extended over several weeks or months. **Oyster BCF:** for tests of standard durations, less than 10% of the organisms in either the dilution water or solvent control treatment showed mortality or adverse sublethal effects. For tests that were extended for several weeks or months, death or other adverse effects were less than 5% per month or did not exceed 30% in all.

» Spawning was not observed; the test treatments did not show evidence of adverse effects [*e.g.*, for oyster BCF studies, excessive mucus production (stringy material floating suspended from oysters), lack of feeding, shell gaping, poor shell closing in response to prodding, or excessive mortality], such that chemical uptake and depuration was likely impacted.

Study Conditions

» **Fish BCF:** the loading was ≤1.0 g of fish (wet weight) per liter per day. **Oyster BCF:** the loading was ≤1 oyster per liter per hour.

» A flow-through (preferred) or renewal study was conducted for fish BCF (flow rate is ≥5 volume replacements/day). For oyster BCF, a flow-through test system was used.

» A dilution water control [and solvent (vehicle) control, when a solvent was used] was included in the test.

» The vehicle concentration was ≤0.1 mL/L. The use of a solvent (vehicle) is not recommended if at all possible.

» A surfactant or dispersant was not used in the preparation of a stock or test solution.

» All test vessels and compartments had the same dimensions and water volumes.

» Treatments were randomly assigned to individual test vessel locations, and individual test organisms were impartially or randomly assigned to test vessels.

» If a precipitate was observed, water was centrifuged and filtered prior to measuring chemical concentration in the water.

» Test vessel aeration was not used (note that it is not recommended).

» Test conditions were adequately described.

» The alkalinity and conductivity were measured at the beginning of each test in the dilution water. The DO, pH, hardness and temperature were measured weekly and at the beginning and end of the test in the dilution water. The particulate matter was measured at least weekly in the test chambers during the test.

» The total organic carbon (TOC) was maintained at ≤2 mg/L. The TOC and DOC in the dilution water were monitored routinely during both the uptake and depuration phases.

» The dissolved oxygen content was >60% of saturation.

» The temperature was maintained at 20±2^{0}C for the oyster BCF study or at the recommended temperature (refer to OCSPP 850.1730) within a range of ±2^{0}C for the fish BCF study.

» A photoperiod regime was selected from 12 h light:12 h dark to 16 h light:8 h dark.

» **Fish BCF:** the pH was maintained between 6.0 and 8.5 and was constant during the test within ±0.5 pH units. **Oyster BCF:** the range of pH was less than 0.8 pH units.

» **Fish BCF:** the water hardness (as $CaCO_3$) generally ranged between 40 and 180 mg/L for freshwater species and for testing with metals, 40 - 50 mg/L.

» **Oyster BCF:** the salinity for natural seawater was >12 ppt with a weekly range <2 ppt or 20%, whichever is higher. On any given day the range was not more than ±2 ppt, and for artificial seawater <2 ppt.

» **Fish BCF:** The exposure phase lasted until steady state was achieved or for a maximum of 28 days; however, this period was extended for certain test substances until steady-state was reached or for a maximum of 60 days.

» **Oyster BCF:** The exposure phase lasted until steady state was achieved or for a minimum of 4 days or for a maximum of 28 days.

» Organisms were depurated until 95% of the mass was depurated or, for a maximum of 56 days for fish BCF studies or 14 days for oyster BCF studies, whichever comes first.

» **Fish BCF:** two concentrations were tested. **Oyster BCF:** a minimum of one concentration was tested; however, if it was desirable to document that the potential to bioconcentrate is independent of the test substance concentration, at least two concentrations, plus appropriate controls were used.

Analysis

» Water was sampled at appropriate intervals. **Fish BCF:** at least duplicate water samples were taken and analyzed per test interval. **Oyster BCF:** triplicate samples were taken and analyzed per interval.

» The concentration of test substance in the test solutions was measured as specified.

» Pesticide concentrations in water and fish were adequately reported.

» **Fish BCF:** The test concentrations of the test substance in the chambers was maintained within ±20% of the mean value during the uptake phase and did not exceed one-tenth the 96-hour LC_{50}, or the higher concentration of the test substance did not exceed about 1% of the acute asymptotic LC_{50}, or was below the chronic NOAEC, or the appropriate endpoint as determined in a previous test or during the range finding test.

» **Oyster BCF:** The test concentration(s) selected did not stress or adversely affect the oysters and was less than one-tenth the IC_{50} or $<IC_{10}$ determined in either the range-finding or 96-h definitive test under the OCSPP 850.1025 guideline (Oyster Acute Toxicity, Shell Deposition) or less than one-tenth the EC_{50} or $<EC_{10}$ determined in the 48-h definitive test under the OCSPP 850.1055 guideline (Bivalve Acute Toxicity, Embryo-Larval Development).

» Fish tissues [*e.g.*, edible (fillet), inedible (viscera), muscle and/or liver] were appropriately analyzed at the various stages of bioconcentration and depuration, if required.

» The extraction scheme was clearly described, including all methodology. Appropriate analytical methods were provided.

» A reasonable attempt was made to identify the parent and all transformation products.

» A reasonable attempt was made, perhaps with a polar and multiple mild non-polar solvent systems, to extract the test compound and its transformation products from the organisms.

» If a radiolabeled test substance was used, the percentage of radioactivity associated with impurities in both water and (fish or oyster) tissue was determined. Additionally, when analysis for parent and transformation products and/or metabolites was conducted, the material balance was adequate (*i.e.*, all the radioactivity in the fish or oyster tissue could be accounted for).

- If the BCF ≥ 500 L/Kg (or 500x) and the compound was radiolabeled, verification was provided to determine whether the radioactivity was associated with the parent chemical or with transformation products. An attempt was made to identify and quantify products representing ≥10% of the total residues in fish or oyster tissues at steady state. Residues of toxicological concern were quantified, even if <10%.
- Detection limits (LOD and LOQ) for all the analytical methods (water, fish or oyster tissue (including both the low and high doses)) were reported.
- Organisms were sampled on at least 5 intervals during uptake and 4 intervals during depuration.

Reporting

- **Fish BCF:** BCF_{SS} and BCF_K were reported (at a minimum for whole fish, optionally for special purposes also on edible and/or non-edible tissues) on a wet weight and on a lipid normalized basis, as well as k_1 and k_2 (and when appropriate, $BCF_{K,G}$, k_G, and k_M). **Oyster BCF:** BCF_{SS} and BCF_K were reported on a wet weight and on a lipid normalized basis, as well as k_1 and k_2.
- Raw measured data and representative chromatographs were provided.

Sample Storage Stability Study Review Guide

This guide is meant to aid in the review of storage stability studies submitted to support field dissipation, field volatility, and ground water monitoring studies submitted under the Office of Chemical Safety and Pollution Prevention (OCSPP) guidelines 835.6100 through 835.8100. The storage stability study review format is based on the format of the Organisation for Economic Co-operation and Development (OECD) Tier II Summaries (T2S)[1]. Reviewers should add to the T2S format any information, statistical calculations, and formatting that will increase their utility to the Agency. In developing study reviews, reviewers should strive to write concisely and to minimize alterations to the T2S format.

Sections I, II, and III of this document provide additional details regarding data, formatting, and issues to consider in developing OCSPP enviornmental fate study reviews. Section I identifies sample storage stability study data and formatting that does not appear in the OECD T2S format and that should be added to USEPA reviews. Although this guide is not intended to strictly prescribe where and how to present the data, an example study review template is provided in Section II for guidance. In general, reviewers should follow the example template, especially for unilateral reviews (*i.e.*, reviews conducted without the participation of other agencies). However, reviewers may modify the template as needed or disregard it in the case of multilateral reviews (*i.e.*, reviews with other agencies participating) in which an alternative format is agreed upon by the participating agencies. Lastly, a list of review considerations (Section III) is provided to help reviewers focus on critical study issues and to identify any common major or minor deficiencies.

[1] A Tier II Summary is an OECD format for study reviews prepared by industry. See OECD Guidance Documents for Pesticide Registration at http://www.oecd.org/chemicalsafety/agriculturalpesticidesandbiocides/oecdguidancedocumentsforpesticideregistration.htm (Accessed Dec. 12, 2012).

Section I. Data to Include in the Storage Stability OECD T2S Template

Reviewers should add the following information to the storage stability T2S template to make it compatible with OCSPP guidelines and increase its utility to the Agency:

First page

- The PC code of the test compound and MRID of the study should be provided in the header and on the first page.

- A statement should be included indicating whether the study was conducted in compliance with FIFRA GLP standards (and if not, how not or why not) and whether signed and dated Data Confidentiality, GLP Compliance, Quality Assurance, and Authenticity Certification statements were provided.

- The study classification and a concise statement of any deficiencies that impacted the classification should be provided on the first page of the review.

- A signature line(s) for the final reviewer(s) should be added to the first page of the review. If this is unacceptable for other agencies participating in a multilateral review, then a cover page with the signature line(s) can be attached to each individual review submitted to EFED's files.

Materials and Methods

- Detection limits should be reported for each analyte.

Results and Discussion

- Degradation half-lives should be reported, if calculable.

Study Deficiencies and Reviewer's Comments

- The Conclusions section should list any deficiencies with the study and any additional salient information; this section may be renamed "Study Deficiencies and Reviewer's Comments." Results and conclusions contained in the Executive Summary should not be repeated in this section.

References

- A References section (Section IV) should be added that lists any literature references other than the study report cited in the study review.

Attachments

- A table (*i.e.*, structure table) of the chemical names, SMILES strings, CAS numbers, and structures of the test compounds should be either referenced as a separate, associated document or attached to the study review. 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.

  For multilateral reviews, chemical names, SMILES strings, structures, and CAS numbers are captured elsewhere in the Monograph[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, the Monograph's structure table should be either referenced as a separate, associated document or attached to each individual review.

- Statistical output files containing calculations (such as statistical analyses and outputs) and graphs that support values reported in the study review should be attached to the review. File names should begin with the same name as that of the individual review. For multilateral reviews, the file naming convention should consistently follow an agreed format.

- A Calculations section may be added that lists equations and defines parameters used to calculate any half-lives. This section is currently placed in Attachment III but may be placed in Section I.B.4.

[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.

Section II. Example Sample Storage Stability Study Review Template

Sample storage stability of [test compound(s)] in [frozen soil, water, *etc.*]

Report:	[Provide full citation. Provide the MRID (first) if the review is unilateral.]
Document No.:	[MRID xxxxxxxx]
Guideline:	Non-guideline [Currently, storage stability studies that support environmental fate studies do not fall under a specific OCSPP guideline. 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 and title.] **Signature:** **Date:** [Type date of signature.]

Executive Summary

The storage stability of [test compound(s)] in frozen [environmental medium or media] was investigated. Untreated samples were collected from the study site(s) at [location(s), state(s)/province(s), country], which was/were studied in [MRID(s)], and spiked with [concentration(s)] of [test compound(s)]. The field spikes were shipped and stored frozen ([temperature] °C) under the same conditions as field samples for [#] intervals of up to [#] days. [Indicate whether substantial degradation of test compound(s) occurred, the duration for which degradation was insubstantial, and any degradation half-life(s) if calculable.] [If this study was not conducted with field spikes in support of a field study, modify the executive summary to reflect the study design and state the purpose of the study, if known.]

I. Material and Methods

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

A. Materials

1. Test Materials [Test compound]
Chemical purity: [percentage (HPLC)]
Batch number: [value]
[Repeat or tabulate the information in this section for multiple analytes.]

B. Study Design

1. Experimental conditions

Untreated samples were collected from the study site(s) at [location(s), state(s)/province(s), country], which was/were studied in [MRID(s)], and spiked with [concentration(s)] of [test compound(s)]. The field spikes were prepared by weighing [mean±s.d. units] of [medium] into individual [type of] bottles. These spikes were shipped and stored frozen ([temperature] °C) under the same conditions as field samples for [#] intervals of up to [#] days after fortification. [Indicate whether the spikes were fortified in the field or in the laboratory and, if in the laboratory, the duration(s) of storage prior to fortification in the previous statement.] The fortification solution(s) contained [concentration(s)] of [test compound(s)] in [concentration(s) of solvent]. Spikes were fortified with [volume] of fortification solution, resulting in a fortification concentration(s) of [concentration] of [test compound(s)].

2. Sampling

[Single or duplicate] spikes were taken for analysis at [list intervals after treatment]. [Alternatively, indicate if spikes were treated at each interval, with a single analysis of all spikes occurring on the final day of fortification.]

3. Analytical procedures

[Briefly describe the analytical procedure for the analyses of spiked samples, including a summary of the extraction and clean up steps, the chromatograph column, mobile phase, and detector, and the detection limits (LOD, LOQ) of each analyte. Provide references for the environmental chemistry method(s) used and its associated independent laboratory validation(s).]

II. Results and Discussion

[Indicate whether substantial degradation of test compound(s) occurred, the duration for which degradation was insubstantial, and any degradation half-life(s) if calculable. Calculate degradation half-lives using single first-order kinetics with non-linear regression of the percentages of the applied against time. Provide associated model parameters (C_0 and k) and statistics (r^2 and p).]

Table 1. Storage stability of [test compound(s)] in [frozen soil, water, *etc.*] expressed as percentage of the applied [Duplicate table as needed for additional test compounds and media.]

[Frozen medium]												
Storage Interval (days)	**[Int. 1]**	**[Int. 1]**	**[Int. 2]**	**[Int. 2]**	**[Int. 3]**	**[Int. 3]**	**[Int. 4]**	**[Int. 4]**	**[Int. 5]**	**[Int. 5]**	**[Int. 6]**	**[Int. 6]**
Replicate Number	**1**	**2**	**1**	**2**	**1**	**2**	**1**	**2**	**1**	**2**	**1**	**2**
[Test compound]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]
[Product 1]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]
[Product 2]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]	[#]

n.d. = not detected, n.a. = not analyzed

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

[Attach a table (*i.e.*, structure table) of the chemical names, SMILES strings, CAS numbers, and structures of the analytes 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 (with columns for %AR left blank). 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. 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.]

Degradate Table.docx

Attachment 2: Statistics Spreadsheets and Graphs

Storage Stability
Study Review Tables

[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.]

[The attached Excel file has two example spreadsheets for results and kinetics calculations.]

Attachment 3: Calculations

Calculations were performed by the reviewer using [indicate program(s) used for calculations] and the following equations. [The following equations are anticipated to reflect the NAFTA kinetics guidance as of March, 2012. If these equations are not current, replace them with the applicable equations from current guidance.]

Single First-Order (SFO) Model

(eq. 1)

where,

C_t = concentration at time t (%)
C_0 = initial concentration (%)
e = Euler's number (-)
k = SFO rate constant of decline (d^{-1})
t = time (d)

The SFO equation is solved [with the Excel Solver] by adjusting C_0 and k to minimize the objective function (S_{SFO}) shown in equation 4.

DT_{50} = natural log (2)/k (eq. 2)

DT_{90} = ln (10)/k (eq. 3)

(eq. 4)

where,

S_{SFO} = objective function of SFO model fit ($\%^2$)
n = number of data points (-)
$C_{model,t}$ = modelled value at time corresponding to $C_{d,t}$ (%)
$C_{d,t}$ = experimental concentration at time t (%)

Section III. Sample Storage Stability Study Review Considerations

This list of considerations is provided to help reviewers assess the acceptability of sample storage stability studies. This list is not a screen or a checklist and is not meant to be attached to the study reviews. Listed considerations carry unequal weight. Evaluate them using best professional judgment. Consider all information from the study and from similar studies to determine whether any deficiencies affect the study classification.

» The test compound was Technical Grade (TGAI) or a Pure Active Ingredient Radiolabeled (PAIRA) compound.

» Sample preparation and fortification was described.

» Storage conditions were specified (temperature, containers, lighting, *etc.*)

» Raw measured data and representative chromatographs were provided.

» Dates of fortification, placement into storage, and analysis were provided.

» The study was conducted for at least the duration of the longest sample storage interval of the test compound(s) in associated field and monitoring studies.

» Appropriate analytical methods were used, with LOQs <2.5% AR for the test compounds.

» Limits of detection and quantitation were reported.

» Half-lives were reported, if calculable.

Prospective Ground Water Monitoring Study Review Guide

This guide is meant to aid in the review of prospective ground water (PGW) monitoring studies submitted under the Office of Chemical Safety and Pollution Prevention (OCSPP) guideline 835.7100. More specifically, this guide is for the review of PGW monitoring study final reports and does not fully address the review of study protocols, progress reports, or other interim reports. The PGW monitoring study review format is loosely based on the format of the Organisation for Economic Co-operation and Development (OECD) Tier II Summaries (T2S)[1], although an OECD T2S format specific to PGW monitoring studies is not available currently. In developing study reviews, reviewers should strive to write concisely and to minimize alterations to the T2S format.

Sections I and II of this document provide details regarding data, formatting, and issues to consider in developing OCSPP environmental fate study reviews. Although this guide is not intended to strictly prescribe where and how to present the data, an example study review template is provided in Section I for guidance. In general, reviewers should follow the example template, especially for unilateral reviews (*i.e.*, reviews conducted without the participation of other agencies). However, reviewers may modify the template as needed or disregard it in the case of multilateral reviews (*i.e.*, reviews with other agencies participating) in which an alternative format is agreed upon by the participating agencies. In Section II of this document, a list of review considerations is provided to help reviewers focus on critical study issues and to identify any common major or minor deficiencies.

[1] A Tier II Summary is an OECD format for study reviews prepared by industry. See OECD Guidance Documents for Pesticide Registration at http://www.oecd.org/chemicalsafety/agriculturalpesticidesandbiocides/oecdguidancedocumentsforpesticideregistration.htm (Accessed Dec. 12, 2012).

Section I. Example Prospective Ground Water Monitoring Study Final Report Review Template

Prospective Ground Water Monitoring of [test compound]

Report:	[Provide full citation. Provide the MRID (first) if the review is unilateral.]
Document No.:	[MRID xxxxxxxx]
Guideline:	OCSPP 835.7100 [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 835.7100.' 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 and title.] **Signature:** **Date:** [Type date of signature.]

Executive Summary

[Registrant name] commissioned a prospective ground water monitoring study designed to evaluate the ground water contamination potential of [analytes] at a vulnerable site in [crop] production in the [region] of the United States. The study site, located in [location], was selected based on leaching vulnerability criteria. The predominant soil series at the site was [soil series]. The study was initiated on [date] and terminated [#] days later.

[#] clusters of two wells each with [#]-foot-long screens were installed on-site. Each well cluster had one shallow well with screen [#] feet below ground and one deep well with screen [#] feet below ground. [#] clusters of four lysimeters each were also installed, each with a [#]-, [#]-, [#]-, and [#]-foot-deep lysimeter. [#] lysimeter cluster(s) and [#] well(s) with a [#]-foot-long screen were also installed on a hydraulically up-gradient control subplot.

[Product name], a [formulation type] containing [#%] [active ingredient] [by weight] was applied in a series of [#] [ground broadcast] applications of [#] lbs a.i./A at [#]-day intervals starting on [date] to total [#] lbs a.i./A applied. [Tracer] was also applied once in order to provide a tracer. Soil samples ([range] feet below surface) were [regularly/irregularly] collected at [#] events up to [#] days after the initial treatment (DAIT). Water samples were collected at [#] events from

lysimeters, ground water wells, and from irrigation source water up to [#] DAIT. Precipitation was supplemented with [overhead center pivot] irrigation to approximate 120% of the normal water inputs (precipitation plus irrigation based on crop water demand or historical rainfall data, whichever is greater) during the study.

Application monitoring cards indicated a cumulative total application of [#] lbs a.i./A ([#]% of the theoretical application rate). [Provide the limits of detection (LOD) for the analytes and tracer. Indicate when the analytes and tracer were first detected at each sampled soil depth, lysimeter depth, and well depth and indicate how leaching rates of the analytes compared to that of the tracer.]

[Provide the maximum concentrations of the analytes in the ground water wells and the intervals at which they occurred. Indicate whether the results were consistent with the expected mobility and (formation and) decline of the analytes. If analytes were not detected, indicate whether the LODs were reasonable.]

I. Study Design

[Provide a brief summary of the study design. The site selection, protocol, and other interim reports may be cited for additional detail.]

[State who commissioned the study and why. State where the study was conducted and briefly discuss why the site(s) was/were chosen.]

A. Site Description

[Cite by MRID any site selection or site characterization reports. Describe the study site(s), including any subplots and the control plot(s) (*e.g.*, plot dimensions, grade, crop history, and pesticide history). Characterize the soil mapping units and component soils within the study site (indicate whether the soils agree with the mapping units and their allowable variation). Characterize the soil horizons, the sources of water inputs (precipitation plus irrigation), and the target water input. State whether the control plot and irrigation pump intake (if any) were hydraulically up-gradient from the treated plots. Describe any nearby surface water features. Summarize the study author's characterization of the site hydrogeology, including the hydraulic conductivity of the vadose and saturated zones, the water table depth and fluctuations, and the ground water flow velocity and mean hydraulic gradient.]

[Indicate the crop(s) grown on the field and provide the planting date(s) and method(s), pesticides applied and dates of application, and harvesting date(s). If available, indicate whether the yield of the crop(s) on the study site was consistent with that of the surrounding fields.]

B. Instrumentation

[Briefly describe and depict the locations of the lysimeters, wells, and meteorological equipment installed at the study site.]

C. Treatment

[Provide relevant information on the studied active ingredient and formulation applied (a brief table of environmental fate properties of the active ingredient may be helpful). Provide the application dates and rates for the active ingredient and the tracer as well as any other pertinent information regarding the application methods.]

D. Sampling

[Briefly describe the sampling intervals and practices for any soil, soil pore water, ground water, irrigation water, tank mix, and application monitoring samples. Also describe the sample transport, storage, and handling conditions. Indicate whether duplicate samples were collected and whether samples were composited. Indicate whether control samples were handled similarly to the other samples. Describe any fortification of field spikes.]

E. Documentation

[Catalog by MRID and date of submission all submitted site selection and site characterization, monitoring plan design, other study protocol, quarterly progress, termination, and final reports and any amendments. Briefly summarize any Agency reviews of these reports, indicating any approvals or waivers. Full details of submitted reports are provided in the References section.]

II. Analysis

A. Moisture Conditions

[Characterize the precipitation, irrigation, and evapotranspiration during the study. Indicate how much water was available for recharge and storage after evapotranspiration. Indicate whether cumulative water inputs (precipitation plus irrigation, *e.g.*, on a monthly scale) were 120% of normal water inputs, based on crop water demand or historical rainfall data (*e.g.*, the 30-year normal mean precipitation), whichever is greater). Indicate whether any substantial runoff events occurred.]

[Summarize the soil moisture during the study, highlighting any sharp increases at lower depths and their causes.]

B. Analytical Methods

[Summarize the analytical methods used in the study, briefly describing the extraction, clean-up, and analysis procedures for each matrix and analyte, and the resulting limits of detection (LOD) and quantitation (LOQ). Indicate whether the environmental chemistry methods (ECM) were successfully validated by an independent laboratory. Indicate whether the methods were validated using the type of medium most difficult to extract for each environmental medium. Provide the MRIDs of the ECM and ILV reports.]

1. Method Recoveries (Laboratory Spikes)

[Describe the blanks, method blanks, and fresh laboratory fortifications prepared and analyzed concurrently with test samples. More specifically, for the laboratory spikes, tabulate the percent recovery per concentration, matrix, and analyte, reporting the mean ± relative standard deviation (RSD), the range, and the sample size. Discuss any poor recoveries and any interference from the blanks.]

2. Storage Stability (Field Spikes)

[Describe the fortifications prepared on-site that were shipped, stored, and analyzed under similar conditions to the test samples. More specifically, tabulate the percent recovery per concentration, matrix, and analyte, reporting the mean ± relative standard deviation (RSD), the range, and the sample size. Discuss any poor recoveries. Indicate the maximum storage length and whether it is comparable to the maximum storage length of test samples. Also summarize any available storage stability data for a relevant matrix that was submitted in a separate study report and provide the MRID.]

III. Results

A. Application Rate Monitoring

[Provide and briefly discuss the results of any tank mix samples and application monitoring samples. Indicate whether the individual and cumulative application rates were verified.]

B. Tracer

[Provide the background concentrations of the tracer (*e.g.*, bromide) in soil prior to treatment. Characterize the leaching of the tracer, indicating the sampling intervals when the tracer was first observed at elevated concentrations and when the tracer was at a maximum concentration at each sampled soil depth, lysimeter depth, and ground water well depth. Provide graphs of the tracer concentrations per time at shallow ground water wells and at deep wells.]

C. Analyte(s)

[Characterize the leaching of the analytes, indicating the sampling intervals when they were first observed at or above the LOD, when they were at a maximum concentration, and when they were last observed at each sampled soil depth, lysimeter depth, and ground water well depth. Indicate how their leaching compared to that of the tracer and to abnormal meteorological events. Indicate whether the results were consistent with the expected mobility and (formation and) decline of the compounds. Provide graphs for each analyte of their concentrations per time at shallow ground water wells and at deep wells (tabulated results are placed in the attached Excel spreadsheet).]

IV. 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.]

V. References [List any references cited in the review, including submitted site selection and site characterization, monitoring plan design, protocol, progress, termination, and final reports and any amendments, providing their MRIDs. Also list any Agency reviews of or responses to these reports, providing their DP barcodes.]

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 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]. 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.]

PGW ROCKS Structure Table Form

[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.

Attachment 2: Statistics Spreadsheets and Graphs

103801 45591606
835.7100 Calcs.xlsx

[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.]

[The attached Excel file has four example spreadsheets, one for each of two analytes, one for the tracer, and one for water inputs.]

Attachment 3: Study Base Map

[Insert the base map from the study report.]

Section II. Prospective Ground Water Monitoring Study Review Considerations

This list of considerations is provided to help reviewers assess the acceptability of prospective ground water monitoring study final reports. This list is not a screen or a checklist and is not meant to be attached to the study reviews. However, well written study reviews contain enough information to determine the status of each of these considerations. Listed considerations carry unequal weight. Evaluate them using best professional judgment. Consider all information from the study and from similar studies to determine whether any deficiencies affect the study classification.

» 'Site selection and site characterization' and 'monitoring plan design' reports were submitted to and approved by the Agency. (Study protocol features such as site selection, site hydrogeology, and analyte selection are evaluated using these reports well before the final report is prepared.)

'Quarterly progress', 'termination', and 'final' reports were submitted to the Agency.

» The study design reflects the approved study protocol, especially if the site is not as vulnerable to leaching or the sampling and analysis methods are not as rigorous as might be expected.

» The test sites were located in the United States or in ecoregions representative of domestic (USA) sites.

» The sites used for this study were clearly shown to have no previous use history involving this or closely related compounds or was clearly shown to contain no background residues or analytical interferences.

» The control plot(s) and any irrigation pump intake were located hydraulically up-gradient from the treated plots.

» A Typical End Use Product (TEP) was used, or an adequate justification was provided for the alternative chosen.

» Test substance was added to the soil at the highest recommended label rate for a single application or the highest recommended rate for each of multiple applications as well as at the minimum retreatment interval, if applicable.

» The site was adequately characterized, including a description of the site topography, soil characteristics, and vadose and saturated zone hydrogeology. Agronomic practices (including irrigation and tillage) and the climate (rainfall frequency, amount, and seasonal distribution) at the site were described.

» Soil taxonomy was adequately characterized using the U.S. Department of Agriculture (USDA) or, for foreign soils, the World Reference Base for Soil Resources (WRB) classification system.

» Soil texture was adequately characterized using the USDA classification system.

» A base map and maps of the topography, surface soils, and potentiometric surface of the water table were provided with sufficient detail.

» 120% of normal water inputs (precipitation plus irrigation based on crop water demand or historical rainfall data, whichever is greater) were provided at the site.

» The number and location of soil cores, lysimeters, and ground water wells were sufficient.

» Duplicate samples were collected and not composited.

» Sampling and irrigation timing and frequency were sufficient to evaluate the movement of the tracer and analytes, with greater frequency near application.

» Soil water content was measured during the study.

» Stability of samples during handling and storage was assured, including for the longest test sample storage duration.

» An independently validated environmental chemistry method(s) was/were used in the study.

» Detection and quantitation limits were reported.

» Raw measured data and representative chromatographs were provided.

» The study was conducted until patterns of transport and decline of the test compound and analyzed degradates were established.

Environmental Chemistry Method (ECM) and Independent Laboratory Validation (ILV) Report Review Guide

This guide is meant to aid in the review of environmental chemistry method (ECM) reports and associated independent laboratory validation (ILV) reports submitted under the Office of Chemical Safety and Pollution Prevention (OCSPP) guideline 850.6100, often in support of field dissipation, field volatility, and ground water monitoring studies (OCSPP guidelines 835.6100 through 835.8100). The ECM/ILV report review format is based on the format of the Organisation for Economic Co-operation and Development (OECD) Tier II Summaries (T2S)[1]. Reviewers should add to the T2S format any information and formatting that will increase their utility to the Agency. In developing reviews, reviewers should strive to write concisely and to minimize alterations to the T2S format.

Sections I, II, and III of this document provide additional details regarding data, formatting, and issues to consider in developing OCSPP ECM/ILV report reviews. Section I identifies data and formatting that does not appear in the OECD T2S format for ECM/ILV report reviews and that should be added to USEPA reviews. Although this guide is not intended to strictly prescribe where and how to present the data, an example ECM/ILV report review template is provided in Section II for guidance. In general, reviewers should follow the example template, especially for unilateral reviews (*i.e.*, reviews conducted without the participation of other agencies). However, reviewers may modify the template as needed or disregard it in the case of multilateral reviews (*i.e.*, reviews with other agencies participating) in which an alternative format is agreed upon by the participating agencies. Lastly, a list of review considerations (Section III) is provided to help reviewers focus on critical ECM/ILV report issues and to identify any common deficiencies.

[1] A Tier II Summary is an OECD format for study reviews prepared by industry. See OECD Guidance Documents for Pesticide Registration at http://www.oecd.org/chemicalsafety/agriculturalpesticidesandbiocides/oecdguidancedocumentsforpesticideregistration.htm (accessed Dec. 12, 2012).

Section I. Data to Include in the ECM/ILV OECD T2S Template

Reviewers should add the following information to the T2S template for analytical methods in soil, water, and air to make it compatible with OCSPP guidelines and increase its utility to the Agency:

First page

- The PC code of the main analyte and MRIDs of the ECM and ILV reports should be provided in the header and on the first page.

- A statement should be included indicating whether the method validations were conducted in compliance with FIFRA GLP standards (and if not, how not or why not) and whether signed and dated Data Confidentiality, GLP Compliance, Quality Assurance, and Authenticity Certification statements were provided.

- The method classification and a concise statement of any deficiencies that impacted the classification should be provided on the first page of the review.

- A signature line(s) for the final reviewer(s) should be added to the first page of the report review. If this is unacceptable for other agencies participating in a multilateral review, then a cover page with the signature line(s) can be attached to each individual review submitted to EFED's files.

- An Executive Summary should be added to the review.

Recovery Findings

- Recovery results from both the initial and independent validations should be tabulated in separate tables. Analytes should be listed in a column to the left. The tables should be expandable to address multiple matrices.

Method Characteristics

- The Linearity, Specificity, Limit of Quantitation, Repeatability, and Reproducibility sections should be merged into a "Method Characteristics" section in which these issues are concisely addressed in a table.

Method Deficiencies and Reviewer's Comments

- The Conclusions section should list any deficiencies with the analytical method, the laboratory validations, and their documentation; this section may be renamed "Method Deficiencies and Reviewer's Comments."

References

- A References section (Section V) should be added that lists any literature references other than the ECM and ILV reports cited in the study review.

Attachments

- A table (*i.e.*, structure table) of the chemical names, SMILES strings, CAS numbers, and structures of the analytes should be either referenced as a separate, associated document or attached to the method review. 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 (with the right four columns blank).

  For multilateral reviews, chemical names, SMILES strings, structures, and CAS numbers are captured elsewhere in the Monograph[2]. Therefore these data are not attached to each method 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.

[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.

Section II. Example ECM/ILV Report Review Template

Analytical method for [analyte(s)] in [soil, water, air, or other environmental medium]

Reports:	ECM: [Provide full citation. Provide the MRID (first) if the review is unilateral.] ILV: [Provide full citation. Provide the MRID (first) if the review is unilateral.]
Document No.:	[MRIDs xxxxxxxx & xxxxxxxx]
Guideline:	850.6100 [U.S.] 8.2.2.1 [soil]; 8.2.2.2 [sediment]; 8.2.2.3 [water]; 8.2.2.4 [biota] [Canada] [If this review is multilateral, also provide the guideline numbers under which participating agencies are reviewing the analytical method.]
Statements:	[Indicate whether the method validations were conducted in compliance with FIFRA GLP standards and whether signed and dated Data Confidentiality, GLP Compliance, Quality Assurance, and Authenticity Certification statements were provided for the method and ILV reports. If the validations were not conducted in compliance with FIFRA GLP standards, indicate why or how they deviated.]
Classification:	This analytical method is classified as [provide classification and very concise statement of any deficiencies that impacted the classification] [E.g.: *"… acceptable for (applicable residues). However, the independent laboratory validated limit of detection for (analyte X) is 10x higher than that stated in the ECM report."*] [If multiple classification terminologies are needed for multilateral reviews, list or tabulate them.]
PC Code:	[xxxxxx]
Reviewer:	[Provide final reviewer(s)'s name and title.] **Signature:** **Date:** [Type date of signature.]

Executive Summary

[This section does not appear in the original T2S template. Table 1 is prepared in the on-line ECM Index[3] format for ease of posting.]

This analytical method, [method ID], is designed for the quantitative determination of [analyte(s)] in [matrix/matrices] using [acronym of chromatograph/detector] (see Table 1). The method is quantitative for [the analytes or a subset thereof] at [the stated LOQ(s) of X µg/L or other value #x higher than the stated LOQ(s)]. The LOQ(s) [is/are] [less than/equal to/greater than] the lowest toxicological level of concern in [matrix/matrices]. [Briefly summarize any major issues discovered by the independent laboratory and state whether the method was modified to address them.]

[3] The ECM Index is found at http://www.epa.gov/pesticides/methods/ecmindex.htm (accessed Nov. 7, 2012).

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
[Pesticide &/or Degradate(s)]	[MRID]	[MRID]	[Leave blank]	[Water/Soil/ Sediment/Plant]	[##/##/##]	[Company Name]	[Acronym of column/ detector]	[#] [µg/L or µg/kg]

I. Principle of the Method

[Briefly describe the analytical method (including any preparation, extraction, cleanup, analyte spiking, derivatization, and analysis steps) and the analytes that the method will quantify. Note whether, for any analytes, the independently validated limits of detection and quantification differ from those of the initial method validation.]

II. Recovery Findings

[Briefly indicate whether mean recoveries and relative standard deviations (RSD) were within guideline requirements (mean 70-120%; RSD ≤20%), *i.e.*, whether the method is quantitative, for each analyte and in each matrix.] [Repeat or expand Tables 2 and 3 for each matrix.]

Table 2. Initial Validation Method Recoveries for Analytes in [Matrix]

Analyte	Fortification Level (units)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
[Analyte x]	[LOQ]	[7]	[#-#]	[#]	[#]	[#]
	[10x LOQ]	[7]	[#-#]	[#]	[#]	[#]

Table 3. Independent Validation Method Recoveries for Analytes in [Matrix]

Analyte	Fortification Level (units)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
[Analyte x]	[LOQ]	[5]	[#-#]	[#]	[#]	[#]
	[10x LOQ]	[5]	[#-#]	[#]	[#]	[#]

III. Method Characteristics

[This section combines the "Linearity," "Specificity," Limit of Quantitation," "Repeatability," and "Reproducibility" sections in the original T2S template.]

[Briefly state how the LOD and LOQ were calculated and whether the calculation procedures are scientifically accepted.] [Provide in Table 4 the limits of quantitation (LOQ) and detection (LOD) established by the independent laboratory validation (ILV). For linearity, provide the correlation coefficient (r^2) and concentration range for the calibration curve. The linearity is

satisfactory when $r^2 \geq 0.995$.[4] State "Yes" where the method is satisfactorily repeatable, reproducible, and specific and provide a short explanation where the method is not. Repeatability is satisfactory when mean recoveries are 70-120% and RSDs are ≤20%. Reproducibility is satisfactory when the independent validation confirms the LOQ(s) established by the initial validation. Specificity is satisfactory when the method includes confirmation of analyte identity and there are no known interferences from the matrix, reagents, solvents, or equipment.]

Table 4. Method Characteristics

	[Analyte x]	**[Analyte y]**	**[Analyte z]**
Limit of Quantitation (LOQ)	[# µg/L]	[# µg/L]	[# µg/L]
Limit of Detection (LOD)	[# µg/L]	[# µg/L]	[# µg/L]
Linearity (calibration curve r^2 and concentration range)	r^2 = [#] [# – # µg/L]	r^2 = [#] [# – # µg/L]	r^2 = [#] [# – # µg/L]
Repeatable	[Yes/No]	[Yes/No]	[Yes/No]
Reproducible	[Yes/No]	[Yes/No]	[Yes/No]
Specific	[Yes/No]	[Yes/No]	[Yes/No]

IV. Method Deficiencies and Reviewer's Comments

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

[List any deficiencies with the analytical method, the laboratory validations, and their documentation. Note whether deficiencies are with the method procedure, the laboratory validations, the documentation, or the method recoveries, and whether they affect the review classification. Some examples of deficiencies are as follows. Use of laboratory equipment that is not commercially available is a deficiency with the method procedure that would not affect the classification. Analyzing only three replicates per concentration and failing to analyze analytes at the LOQ are two deficiencies with the laboratory validation that would affect the method classification. Failing to provide representative chromatographs and failing to explain how the LOD and LOQ were calculated are two deficiencies with the documentation that would affect the method classification. (ECMs with poor documentation may be classified unacceptable even though they are valid and useful in practice.) Mean recoveries outside the range of 70-120% and/or with relative standard deviations greater than 20% typically invalidate a method and would affect the method classification. An LOQ above toxicological levels of concern does not invalidate the method, but results in an unacceptable method classification. If a major issue was discovered by the independent laboratory, then state whether the method was modified to address it and whether a new internal validation was performed.]

[If the initial validation was performed by a governmental agency, a reference to the agency's documentation of the ECM will serve as the ECM report. More specifically, if the applicant submits an ILV report and documentation of the agency's ECM, the initial validation report for the ECM is not needed. If the initial validation was performed by a private entity, the current

[4] This criterion is consistent with Superfund analytical methods for inorganic analytes at http://www.epa.gov/superfund/programs/clp/download/ism/ism1nfg.pdf (accessed Nov. 7, 2012).

applicant needs to submit two reports of performance data, as usual, one for the initial or other internal validation and one for the ILV.]

[Examples of satisfactory method reports, method reports with major deficiencies, and method reports with minor deficiencies are listed below:]

[Satisfactory method reports provide clearly written procedures for sample preparation, extraction, cleanup, derivatization (if required), and analysis. The procedures may be contained in a stand-alone analytical method report with a detailed narrative, a detailed flow chart, or both. The analytical procedures include information on the sample preparation technique, type of instrument and analytical column(s) used, instrument setup and operating parameters, standard and reagent preparation and calibration procedures. Method performance is demonstrated by acceptable recovery data (*i.e.*, method repeatability). Chromatograms for one standard, one matrix blank, and matrix spike at the LOQ and 10 x LOQ spiking levels with response values (*i.e.*, area counts) are included, as well as a regression analysis that defines the slope, intercept, and standard error of the calibration curve. Required equipment and glassware are generally available. An ILV has been performed on the same version of the analytical method and produced acceptable performance data, as described above, which are separate from those of the initial performance data. A report on the ILV findings accompanies the registrant's method report. Analysts seeking to validate a satisfactory method should be able to produce reliable and satisfactory data with minimal interpretation or additional instructions.]

[Method reports may be categorized as having major deficiencies with respect to the analytical procedures and/or performance data. Examples of major deficiencies with a method include lack of detail in the description of the analytical techniques, requirement of equipment or techniques not generally available, use of old or outdated methodologies or obsolete equipment, and lack of repeatability at or above the LOQ. Major deficiencies with a method report include lack of any performance data at the LOQ and 10 x LOQ and lack of an ILV report. Validation of methods with major deficiencies may not be possible without additional information from the registrant.]

[Method reports categorized with minor deficiencies provide adequate procedures for the sample preparation, extraction, cleanup, derivatization (if required), and analysis, but may lack the level of detail provided by satisfactory method reports. Procedures for standard and/or extraction procedures may lack detail and require interpretation on the part of the analyst. Method reports without sufficient performance data (*i.e.*, insufficient number of spiked samples and/or the absence of spikes at the LOQ and 10 x LOQ), sample chromatograms, and/or precision data obtained from sample spikes at the LOQ and 10 x LOQ are considered to have minor deficiencies. Generally, method reports with minor deficiencies will require greater interpretation and professional experience in order to produce acceptable data.]

[For further reference, the following is a list of common deficiencies reported for ECM reports:

- Calibration curves and representative chromatograms/spectra for each analyte measured in each matrix at all spiking levels were not provided.
- Copies were not provided of the chromatograms/spectra for the standards that were used to quantify the analyte(s).

- Example calculations were not provided showing how the raw data were converted to a final concentration.
- A statement was not provided to confirm that the scientists who developed the original ECM differed from those who performed the ILV.
- The method report has conflicting information on the method in different sections of the document. For example, the method report may list different columns in different sections.
- A confirmatory method such as mass spectrometry was not provided to confirm the identity of the compound.]

V. 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 or refer to this table if it exists in a separate, associated document. Do not include 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 (with the right four columns left blank). 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. Therefore these data are not attached to each method 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.]

Degradate Table.docx

Section III. Environmental Chemistry Method Report Review Considerations

This list of considerations is provided to help reviewers assess the acceptability of environmental chemistry method reports and their associated independent laboratory validation reports. This list may be used as a screen or a checklist but is not meant to be attached to the method report reviews. Listed considerations carry unequal weight. Evaluate them using best professional judgment. Consider all information from the method reports and from reports for similar methods to determine whether any deficiencies affect the method report classification.

ECM Report

» The required instrumentation, glassware, and chemicals were identified in the report and are commercially available.

» The matrix/matrices was/were well characterized. (For example, for soil, pH and percentages of organic carbon, moisture, sand, silt, and clay, *etc.* were reported.)

» All steps in the ECM are scientifically sound. Mass spectrometry or another technique was used to confirm the identity of the analyte(s).

» Any encountered interferences, problem areas, or critical steps were described and/or explained.

» The matrix blank was free of interference(s).

» Representative chromatograms were provided for reagent blanks, matrix blanks, standard curves, and spiked samples at the LOQ and 10x LOQ for all analytes in each matrix.

» The chromatograms of the lowest spiking level are attenuated to where one can measure the peak accurately (accounting for the noise on the baseline).

» There are explanations of how the LOD and LOQ were calculated. The procedures are scientifically acceptable. A best effort was demonstrated to achieve a low LOQ. (LOD and LOQ are often calculated as the mean matrix blank value plus 3 times the standard deviation and 10 times the standard deviation, respectively. 40 CFR Part 136, Appendix B lists some scientifically accepted procedures for estimating detection limits. Actual detection limits are not based on the arbitrarily selected lowest concentration in the spiked samples.)

» The LOQ(s) is/are less than toxicological levels of concern. (Concentrations in soil with units of mass/area (*e.g.*, lbs/acre) are converted to units of mass/mass (*e.g.*, mg/kg) using a soil depth of six inches and the soil density. The 6-inch soil depth is a default to use unless there is a reason to use an alternative depth.)

» For ECMs used in submitted field studies, the LOQ(s) is/are less than 10% of the expected or actual peak concentration of the test compound in the field.

ILV Report

» An ILV was performed and documented in a report separate from the ECM report.

» The ILV was independent. (If the laboratory that conducted the validation belonged to the same organization as the originating laboratory, the analysts, study director, equipment, instruments, and supplies of the two laboratories must have been distinct and operated separately and without collusion. The analysts and study director of the ILV must have been unfamiliar with the method both in its development and subsequent use in field studies.)

» All communication prior to running the samples between the independent laboratory and the developers or previous users of the ECM was documented.

» A maximum of three sample sets were used to validate the ECM (*i.e.*, produce recoveries with acceptable precision and accuracy). A minimally complete sample set includes a reagent blank, two matrix blanks, five samples spiked at the LOQ, and five samples spiked at 10x LOQ for each matrix.

» Interferences with peak areas were less than 50% at the LOD.

ECM and ILV Reports

» Sample recoveries were not corrected for reagent blanks, matrix blanks, or other recoveries.

» A minimum of five spiked replicates were analyzed at each concentration (*i.e.*, minimally, the LOQ and 10x LOQ) for each analyte. (For the initial method validation, the number of spiked samples analyzed at each concentration is at least equal to that of the independent method validation and is preferably seven.)

» The method recoveries met OCSPP Guideline 850.6100 criteria for precision and accuracy (mean recoveries for replicates at each spiking level between 70% and 120% and relative standard deviations (RSD) ≤20%) at the stated LOQ and at higher concentrations.

» Two sets of performance data were submitted, one for the initial or other internal validation and one for the ILV, with the following exception. (If the initial validation was performed by a governmental agency, a reference to the agency's documentation of the ECM will serve as the ECM report. In this case, the applicant submitted an ILV report and documentation of the agency's ECM if not the full initial validation report for the ECM.

» Any modifications to the method recommended by the independent laboratory were implemented in the ECM report. If substantial changes to the ECM were recommended, an internal validation was conducted for the updated ECM report.