

PESTICIDE ASSESSMENT GUIDELINES

SUBDIVISION N

CHEMISTRY:

ENVIRONMENTAL FATE

Series 163-1

Leaching and Adsorption/Desorption Studies

ADDENDUM 6 ON DATA REPORTING

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Subdivision N - Leaching and Adsorption/Desorption Studies

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PESTICIDE ASSESSMENT GUIDELINES

ENVIRONMENTAL FATE

Leaching and Adsorption/Desorption Studies

Subdivision N, Section 163-1

DATA REPORTING

INTRODUCTION

A. Purpose

Information from this study enables the Agency to determine the mobilities of the pesticide and its degradates. The information is used to predict the likelihood of the compound and degradates moving in the environment, in particular, the potential for ground water contamination.

B. Objective

This Data Reporting Guideline (DRG) is designed to aid the petitioner/registrant in generating reports which are compatible with the Agency's review process. Data submitters are encouraged to submit complete reports for efficient review by the Agency. PR Notice 86-5, effective on November 1, 1986 (available from the Office of Pesticide Programs, US EPA), pertains to the physical formatting of reports (which are referred to as "studies") and submittal packages. Some of the requirements of PR Notice 86-5 are mandatory.

RESPONSE TO PUBLIC COMMENTS

The purpose of this section is to acknowledge and address the concerns expressed in the letters of comment received by the Agency in response to the public notice in FEDERAL REGISTER, Volume 51, No. 199, p. 36753, Oct. 15, 1986.

This addendum to the Pesticide Assessment Guidelines [Subdivision N] is to be considered an all-encompassing document. The Exposure Assessment Branch (EAB) has reviewed the comments submitted by the registrants and/or committees in regard to the Data Reporting Guideline (DRG). This Guideline is not intended to create new data requirements, but to provide for consistent reporting of the necessary environmental fate data required to perform a scientifically sound hazard assessment. This approach should eliminate most, if not all, of the recycling of submissions between EAB scientists and the registrant.

This discussion has been arranged to consider the general comments concerning environmental fate DRGs first, followed by the specific comments.

General Comments

1. Comment on the location of a specific reporting item:

Considering the need to maintain a consistent format, revisions were made to the DRG in response to these comments wherever possible.

2. Ingredient information from testing laboratories:

It appears some sponsors do not make this information available to testing laboratories. In such a situation, the sponsor bears the responsibility since he is submitting the data for registration.

3. Standard evaluation procedures (SEPs):

SEPs are guidance documents which explain the procedures used to evaluate environmental effects data submitted to the Office of Pesticide Programs. They are also available from the National Technical Information Service. This DRG is compatible with the Agency's review procedures.

4. Appendix(es) - Inclusion of published and previously submitted data:

Previously submitted material, both published and unpublished, may be "resubmitted" by reference to Agency file numbers (e.g. Master Record Identification (MRID) number). In the case of published work which has not been submitted before, it is prudent to submit a copy with the application rather than to assume availability in Agency files.

Study-Specific Comments

1. Comment - TABLE OF CONTENTS:

Should the TABLE OF CONTENTS include the title of each table and figure or simply the page on which each section begins?

Response:

Each table and figure should be listed specifically in the table of contents.

2. Comment:

In Section I.D., please explain what is meant by an aged soil thin-layer study.

Response:

The aging refers to the pesticide in the soil. The test pesticide may be applied to the soil, aged, extracted, and a thin-layer analysis done on the extract. This procedure is intended to identify and characterize both degradates and parent.

3. Comment:

Please explain what an aged adsorption/desorption study is.

Response:

The aging refers to the pesticide in the soil. The test pesticide may be applied to the soil, aged, extracted, and adsorption/desorption testing done on the extract; or, alternatively, applied to the soil, aged, and the batch adsorption/desorption done. This procedure is intended to identify and characterize both degradates and parent.

An aged batch adsorption/desorption study may be used to fulfill the requirement for data on mobility of the aged pesticide, although an aged column leaching study would be acceptable.

4. Comment - Section III:

The use of the word 'narrative' is incorrect and [it should] be deleted or rewritten to convey the intended meaning.

Response:

We disagree, per the definition in Random House Collegiate Dictionary © 1973.

5. Comment - Section III:

[Let] millicuries per millimole (mCi/mmol) and disintegrations per minute per microgram (dpm/ug) replace curies/mole and dpm/g.... The millimole (or micromole) is more representative of the range of concentration used in studies [and] dpm/ug is preferred, as [it] is not only more generically accepted but also routinely used in calculations of pesticide concentration.

Response:

The Agency has no objection to the units this commenter prefers. In any case, units should be clearly specified and used consistently throughout a report.

6. Comment - Analytical Method(s):

The request for method validation, recovery and sensitivity data, quality control procedures and results, would appear to relate to unlabeled (i.e., "cold") methods of analysis involving GLC or HPLC. If so, it should be so stated.

Response:

Method validation, sensitivity, recovery, and quality control are not concepts limited to any specific method(s) of analysis. Unless the method of analysis is a recognized standard method, the applicant

should demonstrate that it actually measures what it is intended to measure.

7. Comment:

Likewise, the inclusion of a "material balance" is not clearly understood and needs further clarification.

Response:

Material balance" is a measure of how completely the starting material (usually, but not always, radiolabelled) is recovered in the analyzed end products. For further details, see Subdivision N of the Pesticide Assessment Guidelines available from NTIS.

8. Comment:

It is suggested that it be noted that figures and/or photographs are generally necessary only when specialized equipment is used.

Response:

The Agency agrees.

9. Comment:

Having a separate heading of TABLES/FIGURES may be confusing. ...[I]t ought to be noted that tables and figures can be presented within the text of the results and discussion or as a separate section.

Response:

Small tables and figures which do not interrupt the flow of the text may be included in the main body of the report. However, extensive tables of data, full page graphs, etc. are preferably placed in a separate section at the end.

GUIDELINE

TITLE/COVER PAGE

Title page and additional documentation requirements (i.e. requirements for data submission and statement of data confidentiality claims) if relevant to the study report must precede the content of the study formatted below. These requirements are described in PR Notice 86-5.

TABLE OF CONTENTS

This page should indicate the overall organization of the study and what material is on which page(s). Tables and figures should be listed in the table of contents.

I. ABSTRACT

This section should contain the overall summary of the study and mention at least the following points:

- A. The soil leaching potential of the pesticide (use the name of the pesticide used throughout the report) was studied by one of the methods cited in the Guidelines (column leaching, soil thin-layer or adsorption/desorption) and indicate whether unaged or aged.
- B. Indicate the site of radiolabeling, if relevant.
- C. If the column leaching method was selected, then indicate the classes of soil studied, how much pesticide was applied to the top of each column (in terms of milligrams and pounds active ingredient per acre), how much water was applied to the top of each column (in terms equivalent to acre inches), the distribution of the pesticide residues through the columns after elution (in terms of percent of applied pesticide per segment of the column) and what percent of the applied was found in the leachate. In this section, also provide the identity of the residues found in the columns and in the leachate in terms of percent of pesticide initially found.

In the aged column study, provide the above plus the initial concentration of the pesticide in the soil that is being aged, the length of the aging period, the weight of the treated, aged soil aliquot taken to overlay the untreated soil column and the concentration of residues in the aged soil aliquot after the aging period.

- D. If the soil thin layer method was selected, then indicate the classes of soil studied, the thickness of the soil layer on the plates, how many micrograms of chemical were spotted on each plate, the distance of the elution front and the calculated R_f values.

In the aged soil thin layer study, provide the above plus the concentration of the chemical in the soil that is being aged, the length of the aging period and the method in applying the aged residues to the untreated soil thin layer plate.

- E. If the adsorption/desorption method was selected, then indicate the classes of soils studied, the concentrations of pesticide studied, and the K_d values obtained.

In the aged adsorption/desorption study, provide the above plus the concentration of the pesticide in the soil that is being aged, the length of the aging period and a brief description of the method of removing the soil aged residues from the soil.

II. INTRODUCTION

This section should open with a description of the purpose of the study, what requirement it is intended to satisfy and (if applicable) how it

supports the position of the registrant. Background and historical information relative to the study should be placed in this section.

III. MATERIALS/METHODS

Materials and methods may be described in separate sections if desired. The instructions below are for a single section combining the two.

This section should be in narrative form. All details with regard to the materials, equipment, experimental design, and procedures used in conducting the study should be placed in this section. If the study was done in several phases, e.g. preliminary ("range-finding") and definitive, describe each phase in detail. In addition, the following are to be included, when appropriate:

A. Chemical

1. The chemical and radiochemical purity of the pesticide, the site of radiolabeling, and the activity of the pesticide in Curies/mole and disintegrations per minute per gram (dpm/gm) or other standard specified units are to be reported here. If non-radiolabeled pesticide was used, the composition of the test material should be reported here.
2. The source, purity and/or composition of the water or solution used for eluting/partitioning is to be reported here.

B. Soils

The soils used in the unaged and aged leaching studies are to be described in this section by listing the following:

1. Percentage of sand, silt, clay;
2. Percentage of organic matter;
3. pH;
4. Cation exchange capacity;
5. Source; and
6. Textural class -- e.g. "sandy loam".

C. Equipment

The description of the experimental design and equipment used should be placed here, especially column design and assembly, the soil thin layer plate material and developing tank, or equipment used in the adsorption/desorption.

D. Test method

1. General

The detailed description of the test method should be placed here. For example:

- a. Any preparation (such as sieving) or modification (such as milling) done to the soil;
- b. How the soil column and soil thin layer plates were prepared;
- c. How the soil was aged for the aging studies;
- d. How the chemical was applied to the soil, the description of the elution or partitioning process, how long elution took or how much time was allowed for soil-water partitioning equilibrium to be reached;
- e. For the column studies, the description of the soil extrusion process, soil column sectioning and other preparations for sampling of both the soil and leachate for analysis;
- f. For the soil thin layer studies, a description of the procedures used for developing the treated soil thin layer plate, visualizing the developed spots on the soil thin layer plate and, if applicable, scraping the spots for analysis;
- g. For the adsorption/desorption studies, a description of how the soil was treated with pesticide, how the partitioning process was initiated and how the soil and water phases were separated and prepared for analysis;
- h. The descriptions of the dates and conditions under which the soil and leachate samples were stored and the thawing procedure (if frozen), in addition to the storage stability data;
- i. How long samples will be retained and under what conditions they will be retained (in case additional work is needed); and
- j. What special problems or difficulties arose during the study that necessitated deviation from the intended test protocol and the effects the deviations had on the results.

2. Analytical Method(s)

- a. The full description of each method used in this study should be placed in this section. This should also include appropriate method validation data, recovery and detection limit data, quality control procedures and results, sample chromatograms, sample calculations and a material balance. The detailed description of the procedures used in preparation and handling of the sample throughout the method should also be placed here. Note that methods for degradation products, when appropriate, are included.

- b. The identity of the instrumentation, equipment and reagents used and the operating conditions of the instrumentation should be placed here.

IV. RESULTS/DISCUSSION

- A. This section should contain the scientific results of the study.
- B. The results of the analysis of the samples are to be placed in this section.
- C. This section also should discuss the leaching potential of the parent compound and, when applicable, the degradative products arising during aging.

V. CONCLUSIONS

This section should contain the discussion of the degree and significance of the leaching potential of the parent compound, and, when applicable, the degradative products.

VI. CERTIFICATION

This should include:

- A. Signatures of each of the senior scientific personnel responsible for the study; and
- B. Certification by the applicant that the report is a complete and unaltered copy of the report provided by the testing facility (except for title page changes required by PR Notice 86-5).

VII. TABLES/FIGURES

- A. Figures/photographs of the equipment used in the methods and flow diagrams of particularly complex extraction/clean-up procedures are to be included here.
- B. This section should contain the table of structures and chemical names/designations for the parent compound and degradative products discussed in the study.
- C. Narrative and tables explaining the steps taken in identifying and quantifying the parent compound and products should be presented here in addition to any graphical presentations of the data (accompanied with the tables of the actual values from which the graphs were constructed).
- D. Tables and figures should be numbered using arabic numerals for figures and roman numerals for tables.

VIII. REFERENCES

IX. APPENDIX(ES)

This is optional. You may attach graphs, raw data, printouts, calculations, and the like, which may further support the study. Reprints of methods and other studies, raw data, copies of relevant letters/memos and material not fitting in any of the other sections should be placed here.