Guidelines for Testing of Chemicals

Draft Proposal for a Test Guideline or Guidance Document

Terrestrial Field Dissipation Studies

Collaborative Preparation

By

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INTRODUCTION

1. Terrestrial field studies of pesticide dissipation demonstrate the transformation, transport, and fate of pesticides under actual use conditions at representative field sites. Laboratory studies on chemical and microbial transformation, leaching, adsorption/ desorption, and volatility are used to develop a conceptual model of the overall dissipation of the pesticide in the field. The field dissipation studies are designed to evaluate the validity of this conceptual model and to integrate the laboratory components of each study. Differences between field study findings and results from the laboratory tests suggest revisions of the conceptual model of pesticide dissipation and possibly the need for additional laboratory and field studies..

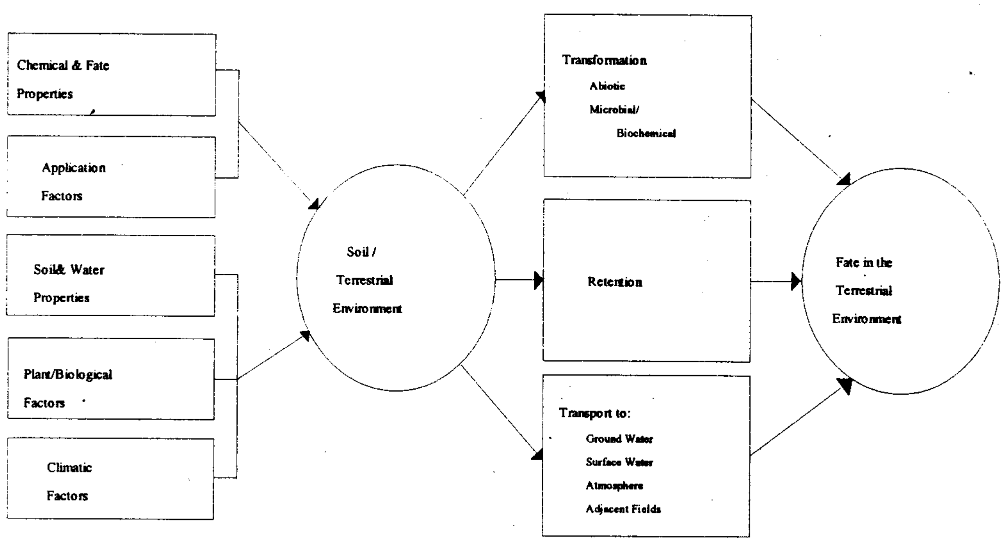

Figure 1. Conceptual Model of the Factors Affecting the Field Dissipation of a Chemical, Adapted From (1).

2. Although the dissipation of a pesticide in the environment commences at the time of discharge from the application equipment, the scope of this guideline is limited to dissipation after the pesticide reaches the plant and soil surfaces of the terrestrial environment. Field dissipation studies do not directly measure spray drift as a route of dissipation.

3. This guideline is based on two national guidelines (2)(3) and two symposia (4)(5).

PRINCIPLE OF THE TEST

4. The study should be designed to identify the route(s) and rate of dissipation of the formulated active ingredient applied under actual field conditions representative of significant areas of pesticide use. It should identify the pathways of transformation, the nature of transformation products, and the distribution of the parent compound and its major transformation products in each environmental compartment. In short, the study should address how the active ingredient changes, which chemical species are formed, and where it ends up. The study should, therefore, be designed to:

   - provide an integrated qualitative and quantitative environmental fate assessment which characterizes the relative importance of each route of dissipation of the parent compound and major and/or toxicologically significant transformation products;
   - confirm whether potential routes of dissipation identified in the laboratory are consistent with field results;
   - characterize the rates of dissipation of the parent compound and formation and decline of the major and/or toxicologically significant transformation products under field conditions;
   - characterize the rates and relative importance of the different transport processes, including leaching, runoff, and volatilization;
   - establish the distribution of the parent compound and the major transformation products in the soil profile;
   - characterize the persistence of the parent compound and major transformation products in soil, including retention and carryover in the soil;
   - characterize foliar dissipation, if the compound is to be applied to plants; and
   - characterize the effect(s) of different pesticide formulations.

APPLICABILITY OF THE TEST

5. Terrestrial field dissipation data are required by regulatory agencies (6)(7) to support the registration of an end-use product intended for outdoor terrestrial uses and to support each application for registration of a manufacturing-use product which may be used legally to make such an end-use product.

6. Terrestrial dissipation studies may be conducted in combination with other field studies such as field residue trials, terrestrial exposure and effects, surface runoff and prospective ground water monitoring studies, provided that all the terrestrial field dissipation study requirements are satisfied. Furthermore, combining terrestrial field dissipation studies with other field studies must be done in a manner that does not compromise the quality of the data produced.

DEFINITIONS AND UNITS

7. Refer to Appendix 1.

INFORMATION ON THE TEST SUBSTANCE

8. The test substance must represent a typical end-use product or a manufacturing-use product that legally could be used to make an end-use product for which terrestrial field dissipation data are required. If the manufacturing use product is usually formulated into end-use products compromising two or more major formulation categories, separate studies must be performed with a typical end-use product for each category.

9. Non-radiolabeled or radiolabeled substances can be used for the test, although non-radiolabeled substances are preferred. The application of radiolabeled substances to field environments is subject to pertinent national and local regulations.

10. The following information on the test substance should be available (see Appendix 2). Such data is important for identifying the potential routes of dissipation to be tracked and, thus, determining the sampling strategies, site locations, sample size and quantity, frequency of sampling. The data is also necessary to interpret the results of the study.

- solubility in water [OECD Guideline 105] (3)(8)(9);
- vapor pressure [OECD Guideline 104] (3)(8)(9);
- Henry's Law constant;
- n-octanol/water partition coefficient [OECD Guideline 107 and/or 117] (3)(8)(9);
- dissociation constant in water, reported as pK_a or pK_b [OECD Guideline 112] (8)(9);
- hydrolysis as a function of pH [OECD Guideline 111] (3)(8)(10)(11);
- photolysis on soil [OECD Guideline in preparation] (3)(10)(12)(13);
- soil aerobic biotransformation [OECD Guideline in preparation] (3)(10)(12)(14);
- soil anaerobic biotransformation [OECD Guideline in preparation] (3)(10)(12);
- adsorption/desorption coefficients [OECD Guideline 106] (3)(10)(12).

11. An appropriate analytical method of known accuracy, precision, and sensitivity for the quantification of the test substance and any relevant transformation products must be available. In most cases, this will require "cold" (i.e., non-radiolabeled) analytical methods that are sufficiently sensitive to track and monitor pesticides residues in the field. The analytical methods are subject to independent laboratory validation (15). See Appendix 3 for a list of considerations in quality assurance/ quality control (QA/QC), reporting and validating analytical methods.

DESCRIPTION OF THE METHOD

Field Plot Systems

12. Plot size must be adequate to demonstrate the transformation, mobility, and fate of the test material in soil under controlled, actual use field conditions. The decision concerning the plot size in field studies should be based on factors such as application rates and methods, crop and management factors, and site characteristics. For pesticides typically applied to cropped or conservation tillage plots (e.g., with at least 30% crop residues on the surface), bareground, pesticide treated control plots are required to help distinguish dissipation pathways.

13. Large-scale studies (21)(22)(23) are conducted using normal agricultural practices (e.g., cultivation prior to planting, etc.) and equipment. The large-scale studies may be used in combination with other field studies, such as crop residue studies, provided the terrestrial field dissipation studies are not disturbed. Small plots (16)(17)(18)(19)(20) are treated using research plot application techniques (e.g., hand-held or backpack sprayers) and, in some cases, may reduce the difficulty in obtaining satisfactory pesticide dissipation curves.

    (a) Large-Scale Studies: Large-scale studies typically cover a treated area of 8 cropped rows by 25 m, but may range to an entire field of several hectares, depending on the design of the experiment and the use for which the product is intended.

    (b) Small Plot Studies: Small plots (i.e., up to 2 m x 2-6 m or 4-12 m^2 in area) are preferable when pesticide dispersion is uneven and dissipation curves may be difficult to generate or interpret.

14. In the case of foliarly-applied pesticides, the test substance should be applied to the intended crop, as specified on the label, in order to characterize foliar dissipation processes. The influence of plant uptake and subsequent foliar dissipation should also be characterized in the case of pre-plant and pre-emergent pesticides as well. When foliar processes interfere with the characterization of soil dissipation processes, a bare study plot (i.e., not sown to intended crops and maintained plant free) should be run in parallel to the cropped study. While the bare plot study may be an artificial system, it is useful to provide an interpretable pesticide dissipation curve in the soil.

<u>Site Selection</u>

15. Field study sites must be representative of the soil, climatic, and management factors under which the pesticide will be used. The factors that should be considered in selecting field study sites include:

    - number of uses/ crops
    - geographic extent and acreage of the crops/ use patterns
    - soil type(s), such as benchmark soils
    - climate (including temperature, amount and distribution of precipitation, solar exposure and intensity)
    - use and management practices

- crop impacts on pesticide dissipation
- pesticide formulation
- timing, frequency, and method of pesticide application
- any label restrictions regarding usage, sites, or conditions

16. Differences in one or more of these factors between the field study sites and the use patterns could affect the fate properties and dissipation processes of the pesticide, thus reducing the applicability of field study results beyond the conditions of the study. Tools, such as a GIS-based decision support model which accounts for the critical factors affecting pesticide dissipation, can be used to determine the most appropriate field sites (24)(25). The GIS decision support model utilizes ecological regions, geospatial soil and agricultural crops databases, and climatic information. Comparable field study area selection is based on environmental conditions and the conceptual pesticide dissipation model identified from laboratory fate studies.

17. Multiple field sites should be used. The actual number of sites needed will depend on a number of factors, including the geographical extent of the use pattern, the number of uses and management practices, and the range in soil and climatic conditions within the geographic extent of the uses.

<u>Field Study Plot Design</u>

18. An assessment of the fate of the pesticide in the terrestrial environment must include all processes that could affect the fate of the chemical, including transformation, leaching, volatilization, runoff, sorption to soil, and plant uptake (1). Terrestrial field studies should be designed, conducted, and evaluated to assess the most probable routes and rates of pesticide dissipation under actual use conditions. The physicochemical properties of the pesticide, the laboratory environmental fate data, application techniques and site characteristics should be considered in designing the field studies. A basic field study design would evaluate field dissipation in soil. If laboratory studies or environmental fate characteristics suggest volatilization, leaching, or runoff are potential dissipation routes, then these components should be incorporated into the study design and sampling scheme.

19. The study design should encompass the range of practices and conditions which reflect the actual usage of the test substance:
    (a) bare soil (noncrop)
        - direct application to the soil surface
        - soil incorporation
        - litter/residue
    (b) cropped, if the use pattern calls for application to crops
        - foliar dissipation, uptake, washoff

20. For all field dissipation studies, noncropped (bareground) plots must be included. If the proposed use pattern includes application of a pesticide on a standing crop, the trial should be conducted with a cropped soil in addition to the noncropped (bareground) plots. The studies should also include an untreated control plots.

21. Because of field scale variability, the experimental units in each terrestrial field dissipation study should be replicated. Replication serves the following functions (26):

- provides an estimate of experimental error;
- improves precision by reducing standard deviation of a mean;
- increases the scope of inference of the experiment by selection and appropriate use of variable experimental units;
- effects control of the error variance; and
- allows statistical comparisons of within-site and among-site variability.

PROCEDURE

Site Characterization

22. Assessing pesticide dissipation requires detailed description of site characteristics as well as characterization of "representative" soils at each test site. Such information is critical to assess *in situ* chemical and physical properties of the test soil.

23. Site description: The study site should be described according to geographic coordinates (ex: latitude, longitude), location on a map (topographic map, aerial photograph or soil survey map), location within the watershed, landforms, landscape position, land surface configuration (slope length and gradient, aspect and direction, micro-relief, roughness, shape, elevation), and depth to ground water. A suggested site description sheet is given in Appendix 4.

24. Soil characterization: At each site, a representative soil pedon should be identified, and a minimum of one soil profile should be described by soil horizons (preferably 2 m in depth) using standard soil morphological properties (depth to and thickness of horizons or layers, Munsell color, texture, structure, macroporosity, depth to a root restricting layer, etc.). Soil profiles will be described and classified to family or series level according to an internationally-recognized system (for example, USDA/NRCS, Canadian or FAO system) representative of the areas where the study is conducted. In addition to the description of soil morphology, information on the soil parent material, vegetation, erosion class, natural drainage class, surface runoff, infiltration, and saturated hydraulic conductivity should be reported. A suggested soil profile description is given in Appendix 5.

25. Soil samples from each horizon should be collected and characterized by determining the physicochemical properties in the laboratory. The physical properties should include particle size distribution (% sand, % silt and % clay, with size fractions specified), textural class (USDA), undisturbed bulk density, and soil moisture characteristic curve (0-15 bar, to help determine the soil water balance throughout the study). The soil chemical properties should include pH, % organic carbon, and cation exchange capacity. Standardized methods should be used and referenced for the determination of these properties. See references (27) and (28) for examples. Depending on the chemical

properties or use site, additional analyses, such as clay mineralogy, specific surface area, and anion exchange capacity (especially in soils dominated by low activity clays or derived from volcanic materials) of the surface soil layer or epipedon and the subjacent horizon (layer) may be helpful for determining sorption potential at the field site. A suggested format for reporting the soil properties is given in Appendix 6.

26. Environmental conditions: Measurement of meteorological variables are necessary to understand pesticide dissipation in the field. Daily records of maximum, minimum, and mean temperature (air and soil), total rainfall, mean wind speed and potential evapotranspiration are required from five days prior to the first application of the pesticide until the conclusion of the study. Where irrigation is used to supplement rainfall, timing and amounts of irrigation water should also be reported. Historical climatological data should be obtained to help evaluate site data with respect to long-term regional variation. Specify the source and location of the historical data. Historical climatic information should include monthly average rainfall, average monthly minimum and maximum temperatures, and the dates and the number of days in the average annual frost-free period. A suggested format for reporting the historical climatic conditions is given in Appendix 7.

27. Management history: Information on the use of the study site, *i.e*., crops grown, pesticides and fertilizers should be provided for the previous three years. The site selected should not have a history of the use of the study pesticide or other pesticides of similar nature (chemical class, common nonvolatile transformation products, etc.) for at least three years prior to the study. This requirement is necessary to reduce analytical interferences and potential microbial adaptations for the test. Management factors such as tillage and cultivation methods, irrigation practices, etc. should be described in detail. A suggested format for reporting the land use and management history is provided in the Appendix 8.

Application of the Test Substance

28. The study should address the effect of pesticide formulation on dissipation. Different formulations are expected to change the fate or transport properties of the pesticide; for example, granular or micro encapsulated formulations may release the active ingredient more slowly than emulsifiable concentrate formulations. For this reason, separate studies should be performed on at least one representative formulation from each of the applicable formulation groups listed below. If the various commercial formulations of a given pesticide are not expected to change the fate of the active ingredient, the applicant should provide the necessary data in support of this assumption within the body of the study report.

29. Recommended groupings of pesticide formulations are:

- water soluble liquids, water soluble powders and emulsifiable concentrates

The release of an active ingredient into the environment is controlled by the formulation type and the site-specific environmental conditions. Water soluble liquids and powders form true solutions when mixed with water and emulsifiable concentrates consist of oil soluble pesticides and emulsifiers. It is expected these formulations will have little effect on the transport of the pesticide in soil (29).

- water dispersible liquids, wettable powders and water dispersible granules

Water dispersible liquids, wettable powders and dispersible granules consist of finely ground solids of various dimensions. Various studies indicate these formulations may affect the transport of pesticides in soil (30)(31)(32). For example, Ghodrati and Jury (30) showed wettable powder formulations may be more resistant to preferential flow than emulsifiable concentrates and technical grade material dissolved in water.

- granules

Granular formulations release the active ingredient gradually as a function of diffusion or leaching resulting from precipitation or irrigation (33). Therefore, this formulation may have a significant effect on transport of the active ingredient if a rain event or irrigation occurs after application.

- micro encapsulated pesticides

Microencapsulated/controlled-release formulations can reduce the potential of leaching through soil (29) but may result in higher surface losses of a chemical when compared to other formulations (34). The available literature on the effect of microencapsulated and controlled-release formulations is inconsistent and testing of this formulation type needs to be evaluated on a case by case basis.

32. The pesticide product shall be applied at the maximum proposed use rate utilizing the same application method(s) as stated on the label. In limited instances (e.g., for ultra-low application rates), it may be necessary to apply the pesticide at a rate greater than the maximum proposed use rate due to analytical detection limits.

33. The pesticide application, including timing and the number of applications, should be consistent with labeling. The pesticide application:

- should occur at the typical time(s) of the year and stage(s) in crop development when it is normally used.
- should be performed according to label instructions for that specific formulation, i.e., a granular typically applied as a band should be applied as a band in the field dissipation study.
- should be incorporated if the pesticide is typically incorporated.
- should be measured by spray cards or similar verification techniques and related to the target application rate and measured concentration in the spray tank.

Where multiple applications are allowed, an experimental design which enables assessments of dissipation from a single application, as well as multiple applications should be used. Replicated treatment plots would evaluate both single and multiple applications.

Study Duration

34. The duration of the terrestrial field dissipation study should be sufficient to determine the DT_{75} of the parent compound and the pattern of formation and decline of major transformation products in the soil, up to a maximum of two years duration. In determining the decline of the major transformation products, the study duration should be sufficient to determine the time required for major transformation products to dissipate to 25% of their maximum detected values in the soil. A major transformation product is one accounting for $\geq$ 10% of the applied at any time during the laboratory studies, or one that has been identified as a potential toxicological or ecological concern.

Management

35. The management (e.g., fertilization, seed bed preparation, weed control, sowing, tillage, harvest) of the field dissipation study site should be carried out in accordance with good agricultural practices. Tillage practices (conventional tillage, conservation tillage, or no-till) should be typical of those used for the particular crop and label recommendations.

36. If the use pattern include irrigation to supplement the water requirements of the plant, then the studies need to be conducted under irrigated conditions. If the use pattern does not involve irrigation, then the field studies do not necessarily have to be conducted with supplemental irrigation. However, if laboratory studies suggest leaching is a potential route of dissipation, then the study must be conducted under conditions in which adequate water is supplied to create leaching conditions.

Environmental Conditions and Monitoring

37. The following environmental conditions should be recorded daily at the study site:

- precipitation
- mean air temperature
- potential evapotranspiration or pan evaporation
- hours of sunshine and intensity of solar radiation
- mean soil temperature
- soil water state.

38. Soil water balance: The soil water content can affect the mode of degradation, degree of microbial activity, potential for volatization, plant growth, and potential for movement (up or down in the soil profile). In order to interpret routes and patterns of dissipation of the test substance, the soil-water content needs to be measured on a regular basis to adequately determine the flux of soil water. Various methods of measuring soil water include tensiometers, time domain reflectometry (TDR), neutron probes, gypsum blocks, and direct measurement of the moisture content of the soil samples (27).

39. Using tracers to track the potential depth of leaching: A conservative tracer can be applied along with the test chemical to help determine the direction, depth, and rate of soil water movement through the vadose zone. Tracer selection should consider the chemistry of the tracer including potential sources of interference, background/baseline levels, analytical detection limits and potential losses such as plant uptake. If a tracer is used, background concentrations need to be analyzed prior to the study.

40. Soil temperature: The soil temperature can also affect the rate of degradation, degree of microbial activity, potential for volatilization, plant growth, and potential for and direction of water movement (up or down in the soil profile). Modern on-site weather stations typically include readily-available measurements of soil temperature, which should be used in interpreting the results of field dissipation studies.

Soil Sampling

41. Soil samples for residue analysis must be representative of each replicate plot at each sampling time. Replicate plots can be defined as repetitive, homogeneous sections of a field treated with the test pesticide in a similar manner to allow comparison between treatments. For example, pesticide treatments could be applied to cropped, bareground, and crop residue sections of the controlled field experiment. Sampling procedures can have a major effect on variability of pesticide concentrations in soil; accurate and consistent sampling is vital for meaningful results. Variables such as plot size, soil variability, crop management practices, pesticide application method, and existing knowledge of the behavior of the pesticide in the environment should be considered in designing an appropriate soil-sampling protocol.

Sampling Patterns

42. A random or systematic soil sampling pattern (35) may be followed, depending on the type of pesticide application and other variables listed above. For example, the soil may be sampled in-row only (seed furrow or band treatment) or by a random pattern which covers the entire treatment area (broadcast application). Great difficulty may be encountered in obtaining interpretable results using an in-row sampling pattern; extreme care should be taken in the application and sampling procedures.

43. In order to avoid variability resulting from possible under-coverage, drift, or edge effects, exclude outside rows of treated areas from sampling.

44. In small plots, systematic sampling is preferred to ensure all treated sectors of the plot are represented and to make it easier to avoid sampling in a previous core hole or in zones where spray patterns in successive passes of the application equipment may have overlapped or failed to cover the surface adequately.

45. To ensure the samples are representative of the entire treated area, an adequate number of cores per plot must be taken at each sampling time - the more the better. The diameter of

a core depends on the number of cores taken, plot size and the type of soil. For example, in a 2.2 x 0.9 m plot, 15 cores (3 cm diameter) should be adequate (16)(17)(36)(37). In large plots, cores of greater diameter are usual, but 10-20 cores will not be sufficient (21)(35). The variability within a large plot is typically greater than in a small plot because of less uniform pesticide application and soil spatial variability.

46. Soil core holes should be marked after sampling. Plugging holes with soil from untreated areas of the site will prevent the cross-contamination at greater depths and subsequent anomalous results.

<u>Depth of Soil Sampling</u>

47. In order to fully demonstrate the fate and transport of the pesticide under study, soil should be collected from a depth sufficient to encompass the vertical distribution of the pesticide and its major transformation products at each sampling time. Data from laboratory studies (physicochemical properties, mobility and transformation) can be used in conjunction with water recharge estimates (e.g., average rainfall data and expected irrigation coupled with evapotranspiration estimates) and soil permeability properties to establish appropriate core depths. Soil sampling should proceed to at least 1 meter depth, particularly for pesticides with laboratory fate characteristics that indicate leaching is an important route of dissipation.

48. The major transformation processes usually occur within the "biologically-active" zone of the soil. For sampling purposes, this zone can be defined as the maximum depth of tillage, rooting depth of agronomic plants, or the depth of an impermeable soil layer, whichever is deepest. If the laboratory studies indicate a low potential of a pesticide to leach, the emphasis of soil sampling designs should be placed on this zone of soil rather than subsoils. The "biologically-active" soil zone concept will allow flexibility in experimental design because of different agronomic practices, types of soil , and site characteristics.

49. For most studies, soil cores should be collected to 1 m in depth and divided into six or more depth increments for analysis (e.g., 15 cm, 15 cm, 15 cm, 15 cm, 20 cm and 20 cm). For low application rate pesticides or where the results of the laboratory studies indicate very low mobility of the parent chemical and its major transformation products in soil, core depths could be sectioned into shorter increments to circumvent dilution of the chemical residues with excess soil. In all cases, analysis of the sectioned cores must clearly define the extent of leaching of the parent chemical and its major transformation products in the soil profile.

50. Soils should be sampled to a sufficient depth such that the lowest section of the sampled cores does not contain detectable amounts of the active ingredient or major transformation products. In the absence of rainfall or irrigation, the initial or zero-time samples can be taken to at least one sample increment below the depth of incorporation.

<u>Times of Soil Sampling</u>

51. Soil sampling should be carried out prior to treatment, immediately after treatment (zero-time) and at increasing intervals (daily, weekly, monthly) between sampling times. Time intervals should be based on the results of laboratory studies and other field studies, if available. Sampling frequency should consider laboratory half-life estimates with increased frequency of sampling for shorter half-life compounds. Compound mobility and site-specific environmental conditions such as rainfall and micro-climate may affect sampling frequency. The frequency of sampling should be concentrated after each application time to characterize the dissipation of the test substance. However, the number and distribution of sample times should also be sufficient to adequately characterize the formation and decline of the transformation products.

52. The determination of the initial concentration in the soil immediately after treatment ("zero-time") is crucial. The pesticide residues in all subsequent soil samples are evaluated in relation to this value and not to the nominal rate of application. An initial residue value that is significantly lower than the value found for a subsequent sample may jeopardize the utility of the study by rendering estimation of decline times (DT_{50}, DT_{75}) meaningless. Duplicate sets of cores should be taken from each replicate plot at the first post-treatment sampling and be processed separately to provide two estimates of the mean initial residue value. An adequate number of the initial samples should be collected to determine significant differences in concentrations during the study period. Initial concentrations should also be determined with appropriate devices such as petri dishes, filter paper or spray cards. In addition, the concentration of active ingredient in the spray tank must be measured immediately prior to and following application. The immediate post-treatment samples should be analyzed as soon as possible after sampling to confirm that the pesticide was uniformly applied to each plot at the intended rate.

53. The dissipation of a product used in multiple applications over a season should be studied through a full cycle of applications (19).

54. Residue data should be obtained until at least 75% of the pesticide and /or its major transformation products have dissipated from the soil profile <u>or</u> the pattern of dissipation has been clearly established (38)(39). It is necessary to determine the 50% decline time (DT_{50}) and DT_{75} from the initial concentration because the dissipation rate constant often decreases with time (i.e., the half-life is not constant as in first-order kinetics).

55. If 75% dissipation is not reached by the time of freeze in the fall, the study should be continued in the following year(s).

56. The plot should be sampled at the end of the growing season to determine residue carryover to the next season (sampling in subsequent years may be necessary). Long-term studies may be required if dissipation is slow to occur. This is particularly important for persistent, low mobility pesticides or those chemicals which show pesticidal activities at low concentrations.

<u>Number and Pooling of Samples</u>

57. The number and diameter (typically 3 to 12 cm) of soil cores to be taken should be based on the size of the plot, the type of soil and the amount of soil required for analysis. Several references for number and diameter of samples are listed in paragraph 49.

58. Corresponding depths of soil cores from a single replicate plot can be pooled and mixed thoroughly to give one representative composite sample from which an aliquot, (e.g., 300 g) can then be taken for analysis.

59. Samples collected from replicate plots should <u>not</u> be pooled or composited across replicates so the within-site variance can be estimated.

60. An adequate number of cores per plot should be collected at each sampling time to ensure the sample is representative of the plot. For example, a composite sample from a 2-m x 1-m small plot may consist of 10 to 15 soil cores (3-cm diameter) per sampling time over a period of one year. For field studies of longer duration, small plots of larger area should be used to accommodate collection of the greater number of cores that would result from an increase in number of sampling times. Owing to the increase in plot size, the number of cores collected per sampling time should be increased.

61. In large plots, soil cores of greater diameter are usual, but 20 or more cores should be collected; the variation present within large plots is greater than that in small plots because of less uniform pesticide application and greater natural variation in the soil.

62. If a large-scale plot contains areas of different types of soil, soil organic matter content, etc., or knolls/depressions, then representative cores from areas of different soil types should be pooled and analyzed separately from other samples (i.e., all samples are <u>not</u> pooled together).

<u>Handling of Samples</u>

63. Soil samples should be frozen if they cannot be extracted immediately.

64. Air-drying of soil samples before extraction is not recommended because of possible loss of chemical residues from the samples via volatilization.

65. To check stability of pesticide residues during storage, untreated soil samples should be fortified with analytical standards (for parent chemical and major transformation products), stored and then extracted and analyzed in the same manner as samples from treated field plots (18). Recovery results from field-fortified samples are preferred to recovery data from more conventional storage stability studies such as laboratory-fortified samples.

<u>Sampling Plants and Foliage</u>

66. Measuring pesticide residues in soil over time provides direct information on a limited

number of dissipation routes, i.e., transformation, sorption and leaching. Other routes of dissipation often play major roles in the environmental fate of the compound. For example, pesticide dissipation may depend upon accumulation and metabolism in plants, volatilization from soil, water, and/or plant surfaces, soil binding, runoff, and spray drift. To meet the objectives of the terrestrial field dissipation study, it may be necessary to design the sampling scheme to account for routes of dissipation which cannot be accounted for through soil core sampling alone.

67. When the pesticide is applied to cropped plots, plant material should be sampled. The sampling scheme should be designed to track the decline in pesticide residues from foliage with time. With foliar application, foliage sampling should include a time zero residue level. Also, pesticide residues may volatilize from foliage more readily than from soil. If volatilization from foliage is likely to be a route of dissipation, the study design must ensure that the appropriate measurements are made. The distribution of sampling times necessary to adequately characterize the dissipation of the test substance from the soil is often inadequate to characterize foliar dissipation. Therefore, samples from foliage need to be collected more frequently at the beginning of the study.

Air Sampling

68. Pesticide residues in the atmosphere demonstrate these compounds volatilize from the field (40). Many pesticides are soil-incorporated, often to retard volatilization and enhance efficacy. In cases where the vapor pressure and Henry’s Law constant of the pesticide or site-specific environmental conditions (e.g., warm temperatures, windy conditions) suggest volatilization may occur following a field application, it is important that the results provide meaningful data on volatilization losses from the field. Therefore, air sampling may be needed to determine whether volatilization is a route of dissipation. The air sampling scheme should be designed to assess volatilization from soil or from plant surfaces. Air sampling methods should be able to measure pesticide residues in vapor phase or on soil or other particles. As with tracking foliar dissipation, air samples need to be collected more frequently at the beginning of the study to adequately characterize the volatilization of the test substance.

Sampling for Pesticide Residues in Runoff

69. Laboratory studies may indicate the potential for pesticide residues to move with runoff water. For these compounds it is important to measure pesticide residues in runoff water and on sediment. This data will help identify a route of dissipation and account for the total amount of pesticide applied to the field. See (41) for guidance on conducting runoff studies.

Sampling strategies to increase sensitivity

70. Sampling strategies that could be used to increase the sensitivity of detecting the pesticide in soil samples include:

- decreasing thickness of sample (thinner increments)
- increasing area of sample
- increasing application rates
- refining/improving analytical methods for parent and major transformation products
- improving recovery efficiencies

DATA ANALYSIS, INTERPRETATION, AND REPORTING

71. Statistical analysis: Data gathered from the study should be analyzed by statistical methods which describe the pesticide's rate of dissipation. Methods should be specified and should be consistent with the study design. The goodness of fit of the data to the statistical analysis should be provided. Analysis should emphasize the dissipation of the pesticide from the upper soil layer to which the pesticide is applied, as well as comparisons of within-site and among-site variability.

72. Data Interpretation and Quantitative Assessment: An evaluation of the data collected in the field dissipation study and interpretation of the results should include the following considerations:

- Half life ($t_{1/2}$) and times for 50 and 75% dissipation of the parent chemical (DT_{50} and DT_{75}, respectively) under field conditions, determined from the residue data;
- Dissipation parameters of the major transformation products (e.g., quantities and rates of formation and decline);
- The mobility of the parent compound and the major transformation products under field conditions;
- A comparison of the dissipation and mobility parameters from the field studies with corresponding results and from laboratory studies and predictions based on the pesticide's physical/chemical properties (e.g., solubility in water, vapor pressure, Henry's Law constant, dissociation constant, octanol/water partitioning coefficient);
- Plant uptake of pesticide residues in the field compared with that under laboratory or greenhouse conditions, within the context of the experimental parameters at the field site, e.g., application, climatic (precipitation and temperature), edaphic (soil properties and moisture conditions) and cropping parameters;
- Identification and discussion of discrepancies between results of the field studies and laboratory studies.

73. Mass Accounting Considerations: The residue data (for the parent chemical, each of the major transformation products and the total major chemical residues) should be expressed in terms of equivalent amounts of parent chemical, and then as percentages of the actual amount of parent chemical initially applied. These percentages can then be summed for the sampled environmental compartments (e.g., soil depths, air, water, plants) and plotted

versus time to estimate an overall mass account. If the overall mass accounting is unexpectedly low, major route(s) of dissipation were possibly not adequately addressed in your field study design.

74. Reporting: The study report should be clear and succinct with definitive conclusions regarding the environmental fate and transport of the pesticide after field application. The study conclusion should be discussed both in terms of the data developed in the field study and in terms of the expected route(s) of dissipation suggested by the laboratory studies. Discussion of how the study compares with other field studies of this active ingredient should be included. The report must clearly identify those aspects of the study having a direct bearing on the study author's conclusions and the validity of the study results.

75. In addition to a full description of the analytical methods used, the following data should be reported:

Information on the test substance and relevant transformation products:
- formulation of the test substance
- limits of analytical detection/quantification
- physicochemical and environmental fate properties
- specific activity and labeling positions (if appropriate)

Information on the field study site:
- location
- climatic conditions and history
- soil taxonomic classification and properties with depth
- hydrologic setting
- size and configuration of the treatment and control plots
- crop, management, and pesticide use history
- depth to the water table

Application of the test substance
- time(s) of application
- rate(s) of application
- method of application
- confirmation of application rate
- field condition at the time of application
- meteorological conditions at the time of application

Tracer(s) used, if any
- type of tracer(s)
- rate and method of application

Maintenance activities:
- type of vegetation
- agricultural practices (date of seeding, time of harvest, yields, etc.)

- weed control

Conditions during test:
- daily air temperature (minimum, maximum)
- daily precipitation and irrigation (reporting of single rainfall events), intensity and duration
- irrigation technique
- weekly and monthly sums of precipitation and irrigation
- weekly mean soil temperature
- soil water content
- daily evapotranspiration or pan evaporation
- movement of tracers (if used)

Residues in soil (as mg/kg dry weight and % of applied amount) at each sample interval:
- concentration of test substance in soil layers
- concentration of transformation products in soil layers
- concentration of extractable radioactivity in soil layers, if applicable
- concentration of non-extractable radioactivity in soil layers, if applicable
- total amounts of test substance, transformation products, other unidentified extractable residues, and non-extractable radioactivity, if appropriate

Residues on and in plants (in mg/kg fresh weight and % of applied amount) at each sample interval, if appropriate

Residues detected via other avenues of dissipation (eg, volatility, runoff, leaching), if appropriate

Mass accounting (recovered percentage of applied test substance) at each sample interval

Appropriate statistical analyses of the collected data

Protocol deviations and amendments

Data should be presented in both tabular and graphical forms.

76. Study Conclusions: The interpretation of the data should identify the major dissipative process(es) operating under field conditions. Field dissipation rates of the parent and pertinent transformation products should be reported. Pathways of transformation (e.g., photolysis, chemical decomposition, biotransformation), transport (e.g., leaching, runoff, volatilization) and biological assimilation (e.g., plant uptake) should be considered. Coupled transport and transformation processes can take place simultaneously. For example, biodegradation can occur during leaching or runoff. Hydrolysis and photolysis can be enhanced by certain soil components in the field that may be absent during laboratory studies. Also, initial products of hydrolysis and photolysis may serve as substrates for microbial degradation. The overall mass account obtained under field

conditions should be discussed in terms of predictions based on laboratory results.

LITERATURE

(1) H.H. Cheng (1990). Pesticides in the Soil Environment: Processes, Impacts, and Modeling. Soil Sci. Soc. Amer. Book Series #2, Madison, WI.

(2) C. Fletcher, S. Hong, C. Eiden, M. Barrett (1989). Standard Evaluation Procedure: Terrestrial Field Dissipation Studies. EPA-540/09-90-073. U.S. EPA Office of Pesticide Programs, Washington, DC.

(3) Agriculture Canada, Environment Canada, and Department of Fisheries and Oceans (1987). Environmental Chemistry and Fate Guidelines for Registration of Pesticides in Canada. T-1-255. Ottawa, Canada.

(4) P.J. Mastradone, J. Breithaupt, P.J. Hannan, J.A. Hetrick, A.W. Jones, R.D. Jones, R.J. Mahler, S. Syslo, J.K. Wolf (1995). Critical assessment of terrestrial field dissipation guidelines. p. 93-98 in *Agrochemical Environmental Fate Studies: State of the Art*, M.L. Leng, E.M.K. Leovey, and P.J. Zubkoff (eds.), Lewis Publishers, Boca Raton, FL.

(5) American Society of Agronomy, Division of Environmental Quality (A5), Symposium “Integrating Environmental Fate and Transport Data from Laboratory to Field Studies.” A.G. Hornsby, Division Chair; J.K. Wolf and J.A. Hetrick, Symposium Chairs. St. Louis, MO, 1995. Abstracts published in Agronomy Abstract (1995).

(6) U.S. Code of Federal Regulations. Part 158 of Title 40, Protection of the Environment.

(7) Canada Pest Control Products Regulations, Section 9.

(8) OECD Guidelines for the Testing of Chemicals (1993). OECD, Paris.

(9) U.S. EPA (1988). Pesticide Assessment Guidelines. Subdivision D: Product Chemistry. U.S. EPA Office of Pesticide Programs, Washington, DC. EPA 540/09-82-018.

(10) U.S. EPA (1982). Pesticide Assessment Guidelines. Subdivision N, Chemistry: Environmental Fate. EPA-540/9-82-021. U.S. EPA Office of Pesticide Programs, Washington, DC.

(11) S.M. Creeger (1985). Standard Evaluation Procedure: Hydrolysis Studies. EPA-540/9-85-013. U.S. EPA Office of Pesticide Programs, Washington, DC.

(12) SETAC-Europe (1995). Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides. Society of Environmental Toxicology and Chemistry.

(13) N.K. Whetzel, S.M. Creeger (1985). Standard Evaluation Procedure: Soil Photolysis

Studies. EPA-540/9-85-016. U.S. EPA Office of Pesticide Programs, Washington, DC.

(14) C.L. Fletcher, S.M. Creeger (1985). Standard Evaluation Procedure: Aerobic Soil Metabolism Studies. EPA-540/9-85-015. U.S. EPA Office of Pesticide Programs, Washington, DC.

(15) D.A. Marlow, D.D. McDaniel, A.E. Dupuy, Jr., E.M. Leovey (1995). Data Reporting Guideline for Environmental Chemistry Methods. Subdivisions N, E, and K. U.S. EPA Office of Pesticide Programs, Washington, DC.

(16) R.A. Chapman, C.R. Harris (1982). Persistence of isofenphos and isazophos in a mineral and an organic soil. J. Environ. Sci. Health B17: 355-361.

(17) C.R. Harris, H.J. Svec, W.W. Sans (1971). Toxicological studies on cutworms. VII. Microplot field experiments on the effectiveness of four experimental insecticides applied as rye cover crop and soil treatments for control of the dark-sided cutworm. J. Econ. Entomol. 64: 493-496.

(18) J. Harvey, Jr (1983). A simple method of evaluating soil breakdown of ^{14}C-pesticides under field conditions. Residue Rev. 85: 149-158.

(19) B.D. Hill (1981). Persistence and distribution of fenvalerate residues in soil under field and laboratory conditions. J. Agric. Food Chem. 29: 107-110.

(20) A. Walker, P.A. Brown (1985). The relative persistence in soil of five acetanilide herbicides. Bull. Environ. Contam. Toxicol. 34: 143-149.

(21) L.A. Birk, F.E.B. Roadhouse (1964). Penetration and persistence in soil of the herbicide atrazine. Can. J. Plant Sci. 44: 21-27.

(22) J.H. Hunter, E.H. Stobbe. 1972. Movement and persistence of picloram in soil. Weed Sci. 20: 486-489.

(23) S.U. Khan, H.A. Hamilton, J.E Hogue (1976). Fonofos residues in an organic soil and vegetable crops following treatment of the soil with the insecticide. Pestic. Sci. 7: 553-558.

(24) D. Kroetsch, R. Gangaraju, W. Effland, I. Nicholson, N. Thurman, and D. Pagurek (1998). A spatial decision support system for the selection of target areas for pesticide field dissipation studies. Paper presented at First International Conference on Geospatial Information in Agriculture and Forestry, 1-3 June 1998, Lake Buena Vista, FL.

(25) Soil Survey and Land Research Centre (SSLRC), Cranfield University (1998). SEISMIC, A unique national spatial information system for environmental modelling. SSLRC, Cranfield University, Silsoe, Bedford, UK. Internet WWW page, URL

<http://www.cranfield.ac.uk/sslrc/projects/seismic/>

(26) Steel, R.G.D. and J.H. Torrie. 1980. Principles and Procedures of Statistics: A Biometrical Approach. McGraw-Hill Book Company, New York, NY.

(27) A. Klute (1986). Methods of Soil Analysis - Part 1: Second Edition, Physical and Mineralogical Methods, American Society of Agronomy, Madison, WI, ASA Monograph No. 9.

(28) A.L. Page (1982). Methods of Soil Analysis - Part 2: Chemical and Microbiological Properties, American Society of Agronomy, Madison, WI, ASA Monograph No. 9.

(29) M. Flury (1996). Experimental evidence of transport of pesticides through field soils - a review. J. Environ. Qual 25:25-45.

(30) M. Ghodrati; W.A. Jury (1992). A field study of the effects of soil structure and irrigation method on preferential flow of pesticides in unsaturated soil. J. Contam. Hydro. 11:101-125.

(31) K. Hurto; M. Prinster (1993). Dissipation of turfgrass foliar dislodgeable residues of chlorpyrifos, DCPA, diazinon, isofenphos, and pendimethalin. p. 86-99. *In* K. Racke, and A. Leslie (eds.) Pesticides in urban environments: fate and significance. American Chemical Society, Washington, DC.

(32) R. Wauchope (1987). Tilted-bed simulation of erosion and chemical runoff from agricultural fields: II. effects of formulation on atrazine runoff. J. Environ. Qual. 16(3):212-216.

(33) C. Furmidge (1984). Formulation and application factors involved in the performance of soil applied pesticides. p. 49-64. *In* R. Hance (ed.) Soils and crop protection chemicals. Monogr. 27. British Crop Protection Council, Croydon, UK.

(34) A. Kenimer, J. Mitchell, A. Felsot, M. Hirschi (1997). Pesticide formulation and application technique effects on surface pesticide losses. Trans. of ASAE 40(6):1617-1622.

(35) F.E.B. Roadhouse, L.A. Birk, 1961. Penetration and persistence in soil of the herbicide 2-chloro-4,6-bis(ethylamino)-s-triazine (simazine). Can. J. Plant Sci. 41: 252-260.

(36) R.A. Chapman, C.R. Harris (1980a). Insecticidal activity and persistence of terbufos, terbufos sulfoxide, and terbufos sulfone in soil. J. Econ. Entomol. 73(4): 536-543.

(37) R.A. Chapman, C.R. Harris (1980b). Persistence of chlorpyrifos in a mineral and an organic soil. J. Environ. Sci. Health B15(l): 39-46.

(38) U.S. EPA (1975). Field dissipation studies. Guidelines for registering pesticides in the United States. 40 FR (123): 26894-26896.

(39) A.E. Smith (1971). Disappearance of triallate from field soils. Weed Sci. 19(5): 536-537.

(40) Taylor, A.W. and W.F. Spencer. 1990. Volatilization and Vapor Transport Processes In H.H. Cheng (ed.) Pesticides in the Soil Environment: Processes, Impacts, and Modeling. Soil Sci. Soc. Amer. Book Series #2, Madison, WI. pp. 213-270.

(41) C.N. Smith, D.S. Brown, J.D. Dean, R.S. Parrish, R.F. Carsel, and A.S. Donigan, Jr. (1985). Field Agricultural Runoff Monitoring (FARM) Manual. EPA 600/3-85/043. U.S. Environmental Protection Agency, Athens, GA.

APPENDIX 1

DEFINITIONS AND UNITS

Plot: a single experimental unit, e.g., a control plot, a treated plot.

Replicate plot: one of two or more plots treated in an identical manner at one site.

Site: exact geographical location of a study.

Major transformation products: degradation products/metabolites of the parent compound that are observed at any time in the laboratory studies at a level greater than 10 percent of the initial concentration of the parent compound. In addition, major transformation products may include other compounds of toxicological significance.

Ideal application and planting techniques: the use of specially adapted application machinery to accurately apply pesticide in small plot field trials in a manner approximating field methods.

Half-life, $t_{1/2}$: The average amount of time required for a concentration of a pesticide to be reduced (i.e., degrade, metabolize, or otherwise dissipate) to one-half of its initial value. With each succeeding half-life period, half of the remaining concentration of pesticide will disappear from the system. The half-life calculation is dependent on the particular reaction model. In the case of a first-order degradation, the half-life assumes a constant rate of degradation.

First-order Kinetics: A model that assumes that the rate of degradation/dissipation is proportional to the concentration of the reactant and remains constant during the reaction time period. In a first order reaction, the plot of the natural logarithm of the concentration of the pesticide versus time will be a straight line. A first-order reaction rate will often (but not always) approximate the degradation of pesticides.

50% Dissipation Time, DT_{50}: The amount of time required for 50% of the initial pesticide concentration to dissipate. Unlike the half-life, the dissipation time does not assume a specific degradation model (e.g., a first-order degradation).

75% Dissipation Time, DT_{75}: The amount of time required for 75% of the initial pesticide concentration to dissipate. Unlike the half-life, the dissipation time does not assume a specific degradation model (e.g., a first-order degradation).

Half-life vs. 50% Dissipation Time: In theory, when the reaction follows a first-order degradation model (the reaction rate is proportional to the reactant concentration and constant over time), the half-life should be equivalent to the 50% dissipation time. However, when the degradation rate is not first-order, the half-life and the 50% dissipation time will differ (normally the half-life will be greater than the 50% dissipation time). Discrepancies between the $t_{1/2}$ and the DT_{50} may suggest that pesticide degradation follows something other than a first-order reaction model.

APPENDIX 2

DATA SHEET TO CHARACTERIZE TEST SUBSTANCE PROPERTIES

This sample data sheet contains the prerequisite information on the test substance physicochemical properties and environmental fate laboratory studies necessary to design the field dissipation study.

Property/lab study	Values	Classification	Reference
Solubility (mg/L)			
Vapor pressure (Pa) Henry's Law Constant (atm/mol/m^3)			
Dissociation constant (pKa or pKb)			
Octanol water partition coefficient (K_{OW})			
Hydrolysis (half-life) Major transformation products			
Soil photolysis (half-life) Major transformation products			
Soil aerobic biotransformation (half-life and persistence) Major transformation products			
Soil anaerobic biotransformation (half-life and persistence) Major transformation products			
Adsorption/desorption (K_d and K_{OC}) Mobility class			
Others			

APPENDIX 3

ANALYTICAL METHOD REPORTING, QA/QC, AND VALIDATION

Example of an environmental chemistry method validation program necessary for the independent validation of analytical methods necessary to conduct field dissipation studies.

Documentation: A full description of the analytical methods used in all steps of the analytical protocol must be submitted, including the following information:

(1) Name and signature, title, organization, address and telephone number of the person(s) responsible for the planning and supervision/monitoring and laboratory procedures/analyses;
(2) Analytical method(s) title/designation/date;
(3) Source of analytical method(s) [e.g. Pesticide Analytical Manual (PAM), Vol. II, scientific literature, company reports];
(4) Principles of the analytical procedure (description);
(5) Copy of the analytical method(s) detailing the following procedures:
    (1) extraction
    (2) clean-up
    (3) derivatization
    (4) determination and calculation of the magnitude of the residue
(6) Reagents or procedural steps requiring special precautions to avoid safety or health hazards
(7) Identification of the chemical species determined
(8) Modifications, if any, to the analytical method(s)
(9) Extraction efficiency
(10) Instrumentation (e.g., GC)
    (1) make/model
    (2) type/specificity of detectors
    (3) column(s) packing materials and size,
    (4) gas carrier and flow rates
    (5) temperatures
    (6) limits of detection and sensitivity
    (7) calibration procedures
(11) Interferences, if any;
(12) Confirmatory techniques
    (1) other column packings,
    (2) detectors
    (3) mass spectrometry
    (4) NMR
(13) Date(s) of sample taking, extraction and residue analyses
(14) Sample identification (coding and labeling information)
(15) Residue results (examples of raw data, laboratory worksheets, stepwise calculation of residue levels, dilution factors, peak heights/areas, method correction factors applied, e.g.

storage stability and method validation recovery values, standard curve(s) used, ppm found of total residues and of individual components if of special concern, range of residue values, representative chromatograms, spectra of control and treated samples);

(16) Statistical treatments of raw data

(17) Other additional information which the registrant/researcher considers appropriate and relevant to provide a complete and thorough description of residue analytical methodology and the means of calculating the residue results.

Quality Assurance/Quality Control: A complete description of the measures taken to ensure the integrity of the analytical results should include information on the following:

(1) Logbooks and/or record keeping procedures, representative instrument printouts, such as chromatograms, spectral analyses, etc.

(2) Sample coding

(3) Use of replicate samples and control blanks

(4) Use of written and validated analytical methodology for residue analyses involved in all test and analytical procedures, including modifications made

(5) Skills of laboratory personnel

(6) Laboratory facilities

(7) Use of high quality glassware, solvents, and test compounds to ensure minimal contamination

(8) Calibration and maintenance of instruments

(9) Good laboratory practices in handling the test substance(s)

(10) Quality assurance project plan

(11) Internal and external auditing schedule established by the study director using an independent quality assurance unit.

Independent Laboratory Method Validation: A full description of the method validation procedures performed by an independent laboratory must be submitted and include the following information:

(1) The recovery level(s) of the test compounds from the soil (substrate) at various fortification level(s) using the residue analytical methodology;

(2) A validated method sensitivity level;

(3) Results of the study and statistical test applied, a stepwise presentation of the procedure for calculating percent recovery from the raw data;

(4) All the data/information necessary to independently verify the results;

(5) Summary of the results; and

(6) Discussion and conclusions of the results.

APPENDIX 4

SITE CHARACTERIZATION DATA SHEET

This sample data sheet can be used to describe the pertinent site characteristics which will influence the dissipation of the test substance in terrestrial environments.

Parameter	Site description	Information Source
Geographic coordinates: Latitude Longitude Method of determination Accuracy of method Data Source FIPS Code for State, County		
Location within watershed		
Landforms		
Landscape position		
Land surface: Slope Gradient Slope Length Direction Micro-relief Roughness Elevation Data Source(s)		
Depth to ground water		
Average rainfall		
Average air temperature: Minimum: Maximum:		
Average soil temperature: Minimum: Maximum:		
Average annual frost-free period: Dates Number of days		
Others		

APPENDIX 5

DESCRIPTION OF THE SOIL PROFILE (USDA)

TAXONOMIC CLASS: Fine-loamy, mixed, thermic Aridic Paleustalfs; Amarillo Series

PEDON DESCRIPTION: Amarillo fine sandy loam--grassland. (Colors are for dry soil unless otherwise stated.)

A--0 to 11 inches; brown (7.5YR 4/4) fine sandy loam, dark brown (7.5YR 3/4) moist; weak fine granular structure; hard, very friable; many fine roots; many fine and medium pores; many wormcasts; mildly alkaline; clear smooth boundary. (5 to 19 inches thick)

Bt--11 to 27 inches; reddish brown (5YR 4/4) sandy clay loam, dark reddish brown (5YR 3/4) moist; moderate coarse prismatic structure parting to weak medium subangular blocky structure; very hard, friable; many fine and medium pores; thin patchy clay films on faces of prisms; clay bridged sand grains throughout; common wormcasts; mildly alkaline; gradual wavy boundary. (8 to 25 inches thick)

Btk1--27 to 38 inches; yellowish red (5YR 4/6) sandy clay loam, moist; weak medium subangular blocky structure; hard, friable; clay bridged sand grains; common films and threads of calcium carbonate on faces of peds; interiors of peds noncalcareous; moderately alkaline; gradual wavy boundary. (8 to 30 inches thick)

Btk2--38 to 56 inches; pink (5YR 7/3) sandy clay loam, light reddish brown (5YR 6/3) moist; weak medium subangular blocky structure; hard, friable; estimated 60 percent calcium carbonate equivalent, 30 percent by volume is concretions of calcium carbonate less than 1 inch in diameter; calcareous, moderately alkaline; gradual wavy boundary. (6 to 36 inches thick)

Btk3--56 to 85 inches; yellowish red (5YR 5/6) sandy clay loam, yellowish red (5YR 4/6) moist; weak very coarse prismatic structure parting to weak medium subangular blocky structure; slightly hard, friable; thin patchy clay films and clay bridging of sand grains; few, mostly vertical stringers of soft bodies of calcium carbonate are concentrated along faces of prisms; few calcium carbonate concretions less than 1 inch in diameter; calcareous, moderately alkaline; gradual wavy boundary. (8 to 50 inches thick)

Btk4--85 to 99 inches; light reddish brown (5YR 5/4) sandy clay loam, yellowish red (5YR 4/5) moist; weak very coarse prismatic structure parting to weak medium subangular blocky structure; hard, friable; thin patchy clay films and bridged sand grains; few soft bodies of calcium carbonate are concentrated in vertical columns along faces of prisms; calcareous, moderately alkaline.

APPENDIX 6

PHYSICOCHEMICAL PROPERTIES OF SOIL

Property	Horizon					Method
Depth						
Texture: %sand %silt %clay Textural class (USDA)						
Bulk density						
Soil moisture characteristic 0 bar 0.1 bar a bar 1 bar 5 bars 10 bars 15 bars						
pH						
Organic carbon (%)						
Cation exchange capacity (meq/100 g) Base saturation (%)						
Clay mineralogy						
Specific Surface						
Anion exchange capacity						
Others						

APPENDIX 7

METEOROLOGICAL HISTORY DATA SHEET

This sample data sheet can be used to describe the pertinent meteorological factors which will influence the dissipation of the test substance in terrestrial environments.

Parameter	**Site description**	**Information Source**
Average monthly rainfall: January February March April May June July August September October November December		
Average minimum/maximum air temperature: January February March April May June July August September October November December		
Average annual frost-free period: Dates Number of days		
Others		

APPENDIX 8

SITE USE AND MANAGEMENT HISTORY FOR THE PREVIOUS THREE YEARS

Use	Previous year	Previous 2nd year	Previous 3rd year
Crops grown			
Pesticide and fertilizer use:			
Cultivation methods: Tillage Irrigation practices			
Others			