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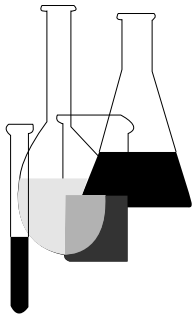
Residue Chemistry Test Guidelines

OPPTS 860.1900 Field Accumulation in Rotational Crops



INTRODUCTION

This guideline is one of a series of test guidelines that have been developed by the Office of Prevention, Pesticides and Toxic Substances, United States Environmental Protection Agency for use in the testing of pesticides and toxic substances, and the development of test data that must be submitted to the Agency for review under Federal regulations.

The Office of Prevention, Pesticides and Toxic Substances (OPPTS) has developed this guideline through a process of harmonization that blended the testing guidance and requirements that existed in the Office of Pollution Prevention and Toxics (OPPT) and appeared in Title 40, Chapter I, Subchapter R of the Code of Federal Regulations (CFR), the Office of Pesticide Programs (OPP) which appeared in publications of the National Technical Information Service (NTIS) and the guidelines published by the Organization for Economic Cooperation and Development (OECD).

The purpose of harmonizing these guidelines into a single set of OPPTS guidelines is to minimize variations among the testing procedures that must be performed to meet the data requirements of the U. S. Environmental Protection Agency under the Toxic Substances Control Act (15 U.S.C. 2601) and the Federal Insecticide, Fungicide and Rodenticide Act (7 U.S.C. 136, *et seq.*).

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OPPTS 860.1900 Field accumulation in rotational crops.

(a) **Scope**—(1) **Applicability.** This guideline is intended to meet testing requirements of both the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) (7 U.S.C. 136, *et seq.*) and the Federal, Food, Drug, and Cosmetic Act (FFDCA) (21 U.S.C. 301, *et seq.*).

(2) **Background.** The source material used in developing this harmonized OPPTS test guideline is OPP 165–2. Field Accumulation Studies on Rotational Crops (Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, EPA Report 540/9–82–021, October 1982). This OPPTS guideline should be used in conjunction with OPPTS 860.1000, Background.

(b) **Purpose.** Data from field accumulation studies on rotational crops will enable the Agency to determine under actual field-use conditions the amount of pesticide residue uptake in rotational crops. Such data are used to establish realistic crop rotation restrictions (time from application to a time in which crops can be rotated) and to provide information for determining whether tolerances are needed in rotational crops.

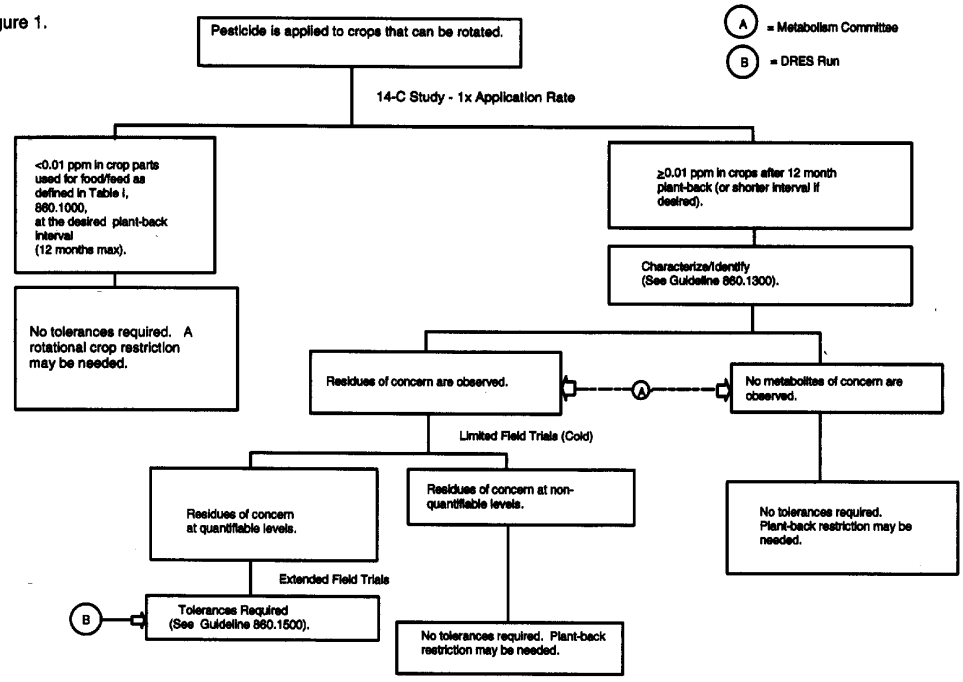
(1) **General considerations.** (i) Studies on confined rotational crops and field rotational crops are conditionally required under 40 CFR part 158 for uses of pesticides on food crops. A rotational crop use is any field-vegetable crop use or any other site use on which it is reasonably foreseeable that any food or feed crop may be planted after harvest of a treated crop. The purpose of field studies is to determine the amount of pesticide residue uptake into rotational crops. The study uses a typical end-product applied to a field plot. Results of these studies are used to determine whether residues occur in rotational crops grown under actual field conditions. Based on these data, appropriate crop rotation restrictions (time from application to planting of rotational crop) may be established and the need for tolerances on the rotated crops determined.

(ii) The Agency has determined that rotational crop studies will not be required for uses of pesticides on the following commodities or crop groups: Asparagus, avocado, banana, berries crop group, citrus fruits crop group, coconut, cranberry, dates, fig, ginseng, globe artichoke, grapes, guava, kiwi fruit, mango, mushrooms, olives, papaya, passion fruit, pineapple, plantain, pome fruits crop group, rhubarb, stone fruits crop group, and tree nuts crop group. Refer to 40 CFR 180.41 for complete lists of the crops in each crop group.

(2) **Scientific considerations.** (i) If the level of the total radioactive residue (TRR) in the confined rotational crops is equal to or exceeds 0.01 parts per million at the desired rotational interval or at 12 months, and once the nature of the residue in the rotational crops is understood, Registrants should consider the Agency's position regarding the residue to be regulated in the primary crop (see OPPTS 860.1850 and the following

Figure 1) to decide whether the first tier of field trials should be initiated. That is, if the composition of the TRR in the rotational crops is such that residues which need to be are regulated are found at levels greater than or equal to 0.01 ppm in the rotational crop (following the criteria set forth in OPPTS 860.1300), field trials should be performed. If residues of concern in the confined study are greater than or equal to 0.01 ppm but less than the limit of quantitation (LOQ) of the analytical method to be used on field trial samples, the Agency will consider waiving the need for field trials on a case-by-case basis.

Figure 1.



(ii) The limited field trials should be conducted on a representative crop (as defined in 40 CFR 180.41) at two sites per crop for the following three crop groups: Root and tuber vegetables, leafy vegetables and small grains (wheat, barley, oats, rye) for a total of six trials. As with confined studies (OPPTS 860.1850), soybeans may be substituted for the leafy vegetable. The six trials should be conducted on crops which a registrant intends to have as rotational crops on the label. If there is no uptake of residues of concern in one or two of the representative crops in the confined study, the Agency still requires six field trials. The trials may be distributed at the petitioner's discretion among the representative crops showing uptake. In addition, some of the six trials could be conducted using other crops that are typically involved in crop rotation such as alfalfa and soybeans.

(iii) The soil should be treated at the *maximum* label rate and the *maximum* number of applications and the appropriate crops should be planted after the *minimum* aging interval. However, if the pesticide is only

to be applied to foliage, the treatment in the field studies may be carried out in the same manner. The crops should be harvested and all the plant parts (including leaves of the root and tuber vegetables) prescribed as raw agricultural commodities (RACs) in Table 1 of OPPTS 860.1000 should be analyzed for the residues of concern observed in primary crops as well as any other residues of concern specific to rotational crops which fulfill the criteria set forth in OPPTS 860.1850. Quantitation limits for rotational crops should be comparable to those for primary crops. The petitioner should describe how the values for the LOQ were calculated and cite any appropriate references.

(iv) The methods employed to analyze the rotational crops should be specific for all pesticide residues of concern in the subject commodities. If the analytical procedure determines interfering compounds it will be considered to be deficient and this would be a cause for rejection of the studies. It would also be desirable to employ sites on which the test pesticide had not been previously applied. If residues are found in control crop samples, the Agency will not automatically reject the field studies. The total study will be examined and consideration given to factors such as the relative levels of residues in treated and control samples. As in the case of the confined accumulation in rotational crops study, analysis of the soil is not required.

(v) If no residues above the LOQ are observed in RACs in the limited field trials, no tolerances will be needed. However plantback restrictions will normally be needed unless the confined study shows no residues of concern at a 30–day plantback interval.

(vi) If the limited field studies in paragraph (b)(2)(ii) of this guideline indicate that quantifiable residues will occur, rotational crop tolerances will be required. The requirement for number of trials would be the same as that to establish primary tolerances on all crops or crop groups which a registrant intends to have as rotational crops on the label. If a registrant desires to allow the universe of crops to be rotated, magnitude of the residue data are required on representative crops (see 40 CFR 180.41) for *all* crop groups which could be planted in a typical crop rotation sequence. With respect to treatment, these trials should be conducted in the same manner as discussed in paragraph (b)(2)(ii) of this guideline for the limited trials. If a registrant believes that fewer crops would be rotated because of the nature of the pesticide or due to the way it is used, guidance should be obtained from the Agency regarding specific data requirements in that case. If tolerances exist on the crops to be rotated as a result of a primary use, rotational data on these crops would be required only if residues in rotated crops are significant in comparison to those in the primary crop.

(c) **Test standards**—(1) **Test substance.** The test substance should be a typical end-use product. If more than one formulation type is registered, several factors need to be considered as to which types should

be used. If one formulation has a significantly higher application rate than the others, it should be applied to the plots in which the rotational crops will be grown. If all formulations have similar application rates but one has been specifically designed to have a longer half-life in the environment (e.g. controlled release product), the latter should be the test substance. The discussion in OPPTS 860.1500 on formulations for crop field trials should also be consulted. That guidance notes that for target crops, residue data can be translated among formulations which are diluted in water and applied early in the growing season. Such translation would also be appropriate for rotational crop field studies. Therefore, rotational crop data reflecting use of a wettable powder would cover formulations such as emulsifiable concentrates and water dispersible granules. However, as with target or primary crops, separate studies will normally be required on rotational crops reflecting uses of granular formulations unless data are available to show the relative soil half-lives of the active ingredient as a function of formulation.

(2) **Test procedure**—(i) **Sites.** Field accumulation studies should be conducted on at least two different sites per crop. These sites should be representative of the areas where rotated crops are expected to be grown. If possible, the soil type at one of the test sites should be the same as that used in the confined accumulation study of OPPTS 860.1850. For restricted use patterns where only one typical area is involved, data from two similar sites should be submitted.

(ii) **Application.** (A) The soil at the test site should be treated with the test substance applied by the method stated in the directions for use specified on the product label and at the highest recommended label rate. However, if the pesticide is only to be applied to foliage, the treatment in the field studies may be done in the same manner.

(B) Following treatment, the pesticide should be aged under aerobic conditions in the soil for a time approximating the anticipated agricultural practice (e.g., 1 year for crops rotated the following year, 120 days for crops rotated immediately after harvest, and 30 days for assessing circumstances of crop failure). Growing a primary crop in the soil during the aging period is not precluded.

(iii) **Sampling.** (A) Representative root and tuber vegetable, small grain, and leafy vegetable crops should be planted as rotational crops. Soybeans may be substituted for the leafy vegetable.

(B) If a registrant is proposing a tolerance for residues in a rotated crop, that crop should be planted, harvested, and analyzed for residues at test sites selected in accordance with the requirements described in detail in OPPTS 860.1500.

(C) The rotational crop RACs (including the foliage of the root and tuber vegetable) as prescribed in Table 1 of OPPTS 860.1000 should be analyzed for residues at appropriate harvest times.

(D) Test duration. Residue data should be collected in rotational crops until the time that the mature portions of the crops are normally harvested.

(d) **Reporting and evaluation of data.** In addition to the applicable reporting requirements specified in OPPTS 860.1000, the following data should be reported:

(1) Field test data including:

(i) Dates of planting and harvesting of primary and rotational crops.

(ii) Amount of rainfall and irrigation water (accumulated from application to harvest).

(iii) Temperature monitoring data and a description of the general climatic conditions at the test site during the study.

(iv) Planting, culture, and harvesting techniques.

(v) Pesticide application dates and method.

(vi) Sampling techniques for primary (if applicable) and rotational crop RACs.

(vii) Stages of crop development at times of sampling.

(viii) Weight of each sample taken for analysis.

(2) Analysis for residues of parent compound and metabolites in the crops. Separate analyses should be conducted on different portions of the plant as outlined in Table 1 of OPPTS guideline 860.1000. In addition, analysis of both the aerial and root portions of root crops should be conducted.

(e) **Format of data report.** The following format is provided as an example. Other formats are acceptable provided that all the information is included.

(1) *Title/cover page.* Title page and additional documentation requirements (i.e. for data submission and statement of data confidentiality of data) if relevant to the study reported should precede the content of the study. These requirements are described in PR Notice 86–5 (see paragraph (f)(2)).

(2) *Table of contents.* The table of contents should follow the title, data confidentiality, and GLP pages. This page should provide the overall organization of the report, including tables and figures.

(3) *Abstract.* Give a summary of the study addressing the following points:

(i) The chemical name and formulation of the pesticide and the method of application to the primary (treated) crop. Structures of the pesticide and metabolites may be included in this section.

(ii) Maintenance of the treated plots.

(iii) A narrative or a table (with an appropriate title) that provides the following information:

(A) Days between treatment and planting of the rotational crops.

(B) Age of crop (in days) at each sampling point (e.g. at forage, hay, grain stages).

(C) Total residues in parts per million. Parent compound and metabolites of concern should be reported separately if so determined by the method.

(iv) Indication of problems (such as technical difficulties or unusual weather) resulting in necessary deviations from the intended test protocol. Describe the effect of the deviations on the results of the study.

(v) The name and phone number of a contact person should be provided in the event the reviewer has technical questions about the study. (This is optional. However, providing this information will facilitate efficient review in case of questions.)

(4) *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 appear in this section.

(5) *Materials/Methods.* This section should be in narrative form in the following order and should contain all details with regard to the materials, equipment, experimental design, field plots and procedures used in conducting the study. Registrants are encouraged to include drawings and photographs of the plot, equipment and of different phases of the study.

(i) *Chemical*. (A) Active ingredient and type of formulation.

(B) Include the percent (by weight) of the active ingredient and for liquid formulations, the weight of the active ingredient per unit of liquid measure.

(ii) *Site*. (A) Include a map of the test plots indicating their location, topography and size, and location and size of the control plots in relation to the test plots; the soil characteristics (percent sand, silt, clay, and organic matter, pH, water holding capacity).

(B) A complete record of daily temperature and daily rainfall throughout the study (refer to raw data discussion in OPPTS 860.1000) and how they compare to average temperature and rainfall at the test site.

(iii) *Crop.* (A) Crop and pesticide use history on the plot for the 3–year period preceding the study.

(B) The date and technique of plot preparation prior to pesticide application.

(C) The identity of the primary (treated) crop; a description of how and when the primary crop was planted; how and when the subject pesticide was applied; the weather (temperature, rainfall, windspeed and direction) and condition of the field at time of application; the formulation of the pesticide applied, adjuvants or other compounds added to the spray/application mixture; the application rate and the application technique. Also, provide a similar description for each of any additional applications made of the subject pesticide. Indicate how much pesticide was applied in comparison to actual use rates; and if application technique differed from label recommendations.

(D) A description of any posttreatment crop maintenance such as use of fertilizers and other pesticides, irrigation (when applied, how much, and source), tilling, weeding, etc.

(iv) *Test method*—(A) *General.* (*1*) Provide the date of harvest of the treated crop; describe what was done to the plot after harvest in preparation for planting of the rotational crops.

(*2*) Provide the identity of the rotational crops planted in the study; a description of the procedure used in planting the rotational crops; and days elapsed between planting of crops and treatment with pesticide; a description of all procedures used in the maintenance of the rotational crops (as done for the treated crop), the sampling/harvest method and number of samples/replicates.

(*3*) Describe handling from the time of taking of the samples until analysis with special attention to the conditions under which the sampled rotational crops were stored and the thawing procedure (if frozen). Determine storage stability of pesticide residues. Provide dates the samples were frozen, thawed, and analyzed.

(*4*) Describe any deviation from the intended test protocol and the effects on the results.

(B) *Analytical method.* (*1*) Describe methods fully (or reference if previously submitted), including method validation data, recovery and method sensitivity data, sample chromatograms, and sample calculations. Preparation and handling of the sample throughout the method should be described in detail. Note that methods for metabolites may also be needed.

(*2*) Identify instrumentation, equipment and reagents used and the operating conditions of the instrumentation. If the extraction/clean-up procedure is complex, a flow diagram should be submitted.

(*3*) Identify all plant fractions analyzed in the study, such as grain, forage, hay, and straw in the case of small grains and root and aerial (leafy) portions in the case of root crops.

(6) *Results/discussion.* (i) This section should contain the scientific results of the study, for instance:

(A) Narrative and tables describing the steps taken in determining the pesticide residues in crop samples. (Any graphical presentations of the data should be accompanied by the tables of the actual values from which the graphs were constructed.)

(B) A table of structures and chemical names/designations for the parent compound and metabolites.

(C) Total residues of concern for all RACs as prescribed in Table 1 of OPPTS 860.1000.

(7) *Conclusion.* Provide discussion as to the significance of the residues (if any) taken up, at what intervals residues are taken up by rotational crops (in which crop fractions and at what levels) and at what interval no quantifiable residues of concern can be expected to be taken up by rotational crops.

(8) *Certification.* Include:

(i) Signatures of each of the senior scientific personnel responsible for the study.

(ii) Certification by registrant that the report is a complete and unaltered copy of the report provided by the testing facility.

(9) *Tables/figures.* Use arabic numerals for figures and roman numerals for tables.

(10) *References.*

(11) *Appendixes.* Reprints of methods and other studies cited, actual results of analyses (raw data), copies of relevant letters and memos and other material not fitting in any of the other sections and that support the registrant's case should be placed in this section.

(f) **References.** The following references should be consulted for additional background material on this test guideline.

(1) Environmental Protection Agency. Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, Field Accumulation

Studies on Rotational Crops, Addendum 1, Series 165–2, EPA Report 540/09–86–149, 1986.

(2) Environmental Protection Agency. Pesticide Regulation Notice PR 86–5, Standard Format for Data Submitted under the FIFRA and Certain Provisions of the Federal Food, Drug, and Cosmetic Act (FFDCA), May 3, l986.

(3) Environmental Protection Agency. Pesticide Rejection Rate Analysis, Residue Chemistry/Environmental Fate: Followup Guidance for Conducting Rotational Crop Studies, EPA Report No. 738–B–93–001, February 1993.