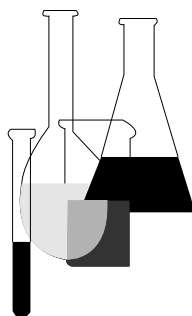




Residue Chemistry Test Guidelines

OPPTS 860.1360 Multiresidue Method



“Public Draft”

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.*).

Public Draft Access Information: This draft guideline is part of a series of related harmonized guidelines that need to be considered as a unit. *For copies:* These guidelines are available electronically from the EPA Public Access Gopher (gopher.epa.gov) under the heading “Environmental Test Methods and Guidelines” or in paper by contacting the OPP Public Docket at (703) 305-5805 or by e-mail: guidelines@epamail.epa.gov.

To Submit Comments: Interested persons are invited to submit comments. By mail: Public Docket and Freedom of Information Section, Office of Pesticide Programs, Field Operations Division (7506C), Environmental Protection Agency, 401 M St. SW., Washington, DC 20460. In person: bring to: Rm. 1132, Crystal Mall #2, 1921 Jefferson Davis Highway, Arlington, VA. Comments may also be submitted electronically by sending electronic mail (e-mail) to: guidelines@epamail.epa.gov.

Final Guideline Release: This document is available from the U.S. Government Printing Office, Washington, DC 20402 on *The Federal Bulletin Board*. By modem dial 202-512-1387, telnet: federal.bbs.gpo.gov 3001, or call 202-512-1530 for disks or paper copies. This guideline is available in ASCII and PDF (portable document format).

OPPTS 860.1360 Multiresidue method.

(a) Scope.

(1) **Applicability.** This guideline is intended to meet testing requirements of 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 guideline 171-4m (see reference in paragraph (d)(1) of this guideline). This OPPTS guideline should be used in conjunction with OPPTS guideline 860.1000, Background, which provides general information and overall guidance for the 860 series on Residue Chemistry.

(b) **Purpose.** Analytical methods capable of determining many pesticide residues in a single analysis have been developed by the Food and Drug Administration (FDA). In order to assess the incidence of the residues remaining on foods and feeds, the Environmental Protection Agency (EPA) uses the data compiled by FDA employing these multiresidue methods in its residue enforcement programs. By using these data FDA chemists can confirm the presence or absence of many pesticides and their metabolites in commodities, and identify many “unidentified analytical responses (UARs)” in samples of unknown treatment history.

(c) Method.

Specific directions for each multiresidue method used by FDA are published in that Agency’s Pesticide Analytical Manual, Vol. I (PAM I)(see paragraph (d)(2)). Compilation of data on the analytical behavior of pesticides and related chemicals are also published in PAM I. The data compiled in this way include: relative retention times of the compounds on a variety of gas-liquid chromatographic (GLC) columns; responses of various GLC detectors to the compounds; recovery of the compound through complete methods, and sometimes through important steps within the methods. The large amount of effort spent on the testing of multiresidue methods and compilation of results is justified by the advantages such compilations offer the analytical chemist. When analytical behavior for numerous compounds through the method in use is known, the analyst is better equipped to recognize the residues that are present in samples of unknown treatment history. In situations where the likelihood of some particular residue is known, the data lists for several methods can be consulted to help choose which method should be used.

An updated compilation of multiresidue methods is provided in Appendix I of PAM I. All petitioners/ registrants are expected to provide recovery data for the methods used. They are expected to follow the directions for the protocols found in PAM I, Appendix II, starting with the “Decision Tree for Multiresidue Methods Testing” and the accompanying guidance

found in the “Suggestions for Producing Quality Data”; i.e. “Decisions on What Protocols to Follow” and “Proper Application of Methods.” decision tree indicates that recovery is likely, then the registrant should consult the “Data Development” section of the proper protocol(s) and precisely follow the guidance offered to generate quality data. It is imperative that all laboratories generating multiresidue methods recovery data follow the directions as written so that EPA can determine how a chemical behaves when analyzed according to a precisely defined method. When data have been generated, the registrants are to use the reporting forms found in PAM I, Appendix II for presenting these data to the Agency. From the completed reports the appropriate recovery data will be extracted and incorporated for a future up-date of Appendix I.

If the recovery is considered to be complete through any of the protocols, then registrants are encouraged to use that protocol as their primary enforcement method. However, registrants need to develop a separate single analyte confirmatory method for PAM II.

(d) **References.** The source material for this guideline is taken directly from the following set of documents.

(1) U.S. Environmental Protection Agency, Pesticide Assessment Guidelines, Subdivision O, Residue Chemistry. EPA Report No. 540/9-82-023, October, 1982.(Available from National Technical Information Service, Springfield, VA)

(2) Pesticide Analytical Manual (PAM), Vols I and II, 1994, Food and Drug Administration, Washington, D.C. (available from National Technical Information Service, Springfield, VA).