United States Environmental Protection Agency Prevention, Pesticides and Toxic Substances (7101) EPA 712–C–96–176 August 1996



Residue Chemistry Test Guidelines

OPPTS 860.1360 Multiresidue Method



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 860.1360 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 guideline is OPP 171–4 Results of Tests on the Amount of Residue Remaining, Including a Description of the Analytical Method Used (Pesticide Assessment Guidelines, Subdivision O: Residue Chemistry., EPA Report 540/9–82–023, October 1982). This OPPTS guideline should be used in conjunction with OPPTS 860.1000, Background.

(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, EPA uses the monitoring data compiled by FDA employing these multiresidue methods in its residue enforcement programs. By using the data generated under this guideline, 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. (1) 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)(1) of this guideline). A compilation of data on the analytical behavior of pesticides and related chemicals is 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 response for numerous compounds 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.

(2) An updated compilation of multiresidue methods is provided in Appendix I of PAM I under paragraph (d)(1) of this guideline. All petitioners/registrants are expected to provide recovery data for parent pesticide and all metabolites of concern through these methods. They are expected to follow the directions for the protocols found in PAM I, Appendix II under paragraph (d)(1) of this guideline, 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. If the decision tree indicates that recovery is likely, the registrant should consult the data development section of the proper protocols and follow the guidance offered precisely 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.

(3) If the recovery is considered to be complete through any of the multiresidue protocols, registrants are encouraged to use that protocol as their primary enforcement method. However, registrants need to develop a separate single analyte confirmatory method for publication in PAM II (under paragraph (d)(2) of this guideline).

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

(1) Pesticide Analytical Manual (PAM), Volume I. Food and Drug Administration, Washington, DC. Available from National Technical Information Service, Springfield, VA (1994).

(2) Pesticide Analytical Manual (PAM), Volume II. Food and Drug Administration, Washington, DC. Available from National Technical Information Service, Springfield, VA (1994).