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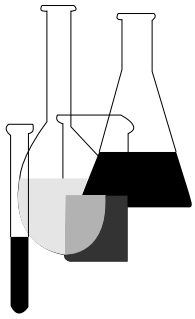
Residue Chemistry Test Guidelines

OPPTS 860.1340 Residue Analytical 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 860.1340 Residue analytical method.

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

(b) **Purpose.** (1) Based on plant and animal metabolism study results, the Agency requires tolerance petitioners to develop analytical methods to determine all components of the total toxic residue (TTR). In some cases, it is not possible to develop a single method that can determine all components of the residue, and several methods are required. Residue analytical methods are used to obtain residue data on which dietary exposure assessments and tolerances are based, and to enforce the tolerance after it is established. Enforcement methods are validated by an independent laboratory before submission to the Agency as required by PR Notice 96–1 (see paragraphs (c)(6) and (e)(3) of this guideline). The Agency validates each new analytical method using a method trial to ensure that the procedures are appropriate for tolerance enforcement.

(2) The methods for residue analyses should serve two functions: They must provide the residue data upon which judgements are made as to the identity and magnitude of residues resulting from the proposed use, and they must provide a means for enforcement of the tolerance. The methods described in the FDA Pesticide Analytical Manual (PAM), Volume II, and the Official Methods of Analysis of the Association of Official Analytical Chemists (as referenced in paragraphs (e)(7) and (e)(1) of this guideline) can be used as examples of suitable analytical methods.

(c) **Test method—(1) General.** (i) The analytical methods must be described in a stepwise fashion in sufficient detail to enable competent analysts to apply the method even though they are unfamiliar with the procedure. Residue analytical methods should be practical, rapid, and quantitate the TTR in the tolerance expression. While the Agency, on a case-by-case basis, may accept best-available methods for the TTR that require state-of-the-art equipment, the equipment must be commercially available in the United States. Reprints of published methods may be submitted. However, where modifications have been made to adapt a basic method to other crops for which a tolerance is proposed, details of the modifications are needed. This includes application to processed byproducts and meat, milk, poultry, or eggs, if these are a consideration.

(ii) The method should not be subject to substrate-related interferences or those arising from reagents. Appropriate clean-up measures should be incorporated to reduce or eliminate spurious responses that might jeopardize the results. For example, in gas-liquid chromatographic (GLC) methods, separation should be sufficiently distinct to yield reasonably discrete peaks for the components of interest rather than a response which appears as a shoulder on an interfering peak.

(iii) The Agency encourages submission of more direct and easily performed methods for tolerance enforcement. However, the subject methods must meet the Agency's stringent criteria for enforcement procedures (see paragraph (c)(5) of this guideline). Although methods used solely for data collection do not need to meet all the requirements of enforcement methods, they must be validated in a similar manner to assure the Agency they are adequate for measuring the TTR.

(iv) The use of the FDA multiresidue methods (MRMs) in PAM, Volume I, under paragraph (e)(6) of this guideline, as primary enforcement methods is encouraged. Petitioners are required to submit MRM test data for the parent compound and all regulated metabolites (see OPPTS 860.1360). It is recommended that petitioners determine the suitability of the MRMs for measuring a new pesticide prior to developing a single-analyte method. If one of the MRMs is found to be acceptable as the enforcement method, an independent laboratory validation as described in paragraph (c)(6) of this guideline will not be required. However, the petitioner will still be expected to provide a single analyte confirmatory method to be published in PAM, Volume II (see paragraph (e)(7) of this guideline).

(v) Whenever possible, GLC retention times and response values should be reported relative to those of a stable reference compound, particularly when the residue is converted to a derivative prior to gas chromatography. GLC and high-performance liquid chromatography (HPLC) parameters should be reported in a form such as the guidelines outlined for the multiresidue method (MRM) protocols found under paragraph (e)(6) of this guideline.

(2) **Validation of method by petitioner.** (i) Methods must be validated by control sample data and recovery data for all components of the TTR on an adequate representation of the commodities involved. Control values should be reasonably low in relation to the proposed tolerance, preferably less than 20 percent of the proposed tolerance. Recoveries should be at fortification levels appropriate to the proposed tolerance and should include the limit of quantitation (LOQ). Recoveries should lie between 70 percent and 120 percent of the known quantity of the pesticide and its metabolites spiked into the substrate controls, and should not vary significantly from sample to sample. Methods in which recoveries are consistently greater than 100 percent are considered questionable. If 70 percent

recovery is not attainable, the Agency will accept, on a case-by-case basis, methods having lower recoveries for active ingredients that are not acutely toxic or for minor metabolites.

(ii) Petitioners are to report individual values for recoveries, standard deviations, and confidence limits for all components of the TTR. The residue levels being measured is a major factor in determining the acceptable variability of a method. Appropriate coefficients of variation (CVs) or relative standard deviations (RSDs) as a function of residue level are discussed in references such as paragraph (e)(5) of this guideline. The Agency will consider the variability in recovery values when determining the acceptability of methods with recoveries outside the 70–120 percent range. For example, a method with average recovery of 65 percent and a low CV (e.g., 5 percent) may be viewed more favorably than a method showing 95 percent average recovery and a CV greater than 20 percent.

(iii) The raw agricultural commodity (RAC), or a macerate thereof, should be fortified, rather than crop, animal tissue, milk, or egg extracts. The portion of the crop to be analyzed is specified under paragraph (e)(6) of this guideline, in 40 CFR 180.1, and in Table 1 in OPPTS 860.1000. Petitioners are also advised to consult the proposed regulation (40 CFR 180.45) on portion of food commodities to be analyzed (see (e)(6) of this guideline). The petitioner should state the estimates of the practical limits of detection (LOD) and quantitation as applied to each of the subject crops or tissues. The estimates of the practical LOD and LOQ should be based on the least concentration of pesticide which can be detected or quantitated with a reasonable degree of assurance, taking into account the size and variation of blanks (instrument response due to crop extractives and reagents). The petitioner should describe how the values for LOD and LOQ were calculated and cite any appropriate references.

(iv) The analytical method should be validated on each crop for which residue data are generated and a tolerance is proposed. In the case of crop group tolerances (e.g. root and tuber vegetables, leafy vegetables (except Brassica vegetables), and cereal grains), the method needs to be validated on only the representative crops for the group as specified in 40 CFR 180.41. The report submitted on the method itself needs to include recovery data on only a representative number of crops. However, in crop field trial reports, additional validation data should be provided on any crop that was not tested for the analytical method report.

(v) With respect to animal commodities, validation data are required for milk, eggs, and all tissues for which residue data are collected in feeding studies and/or for which tolerances need to be established. The tissues normally include cattle muscle, fat, liver, and kidney and poultry muscle, fat, and liver. The recovery data for cattle commodities will in most cases cover the products of goats, hogs, horses, and sheep.

(3) **Extraction efficiency.** (i) Conventional recovery experiments as discussed under paragraphs (c)(2) and (c)(3) of this guideline do not necessarily reflect the efficiency with which aged (weathered) residues are extracted from crops. There should be some assurance that aged residues are completely extracted by the procedure. Utilization of radiolabeled samples from the plant and/or livestock metabolism studies (referred to as radiovalidation of the method) provides the best evidence on completeness of extraction. Analytical methods should be radiovalidated to determine whether the TTR is extracted from weathered plant matrices and/or livestock tissues, milk, or eggs. Samples should undergo the extraction procedure employed in the method. The petitioner needs to demonstrate that the extracted radioactivity accounts for most of the TTR that was identified in the metabolism study. This may involve performing the determinative step of the method or other procedures which will identify and quantitate the components of the extracted radioactivity. Simply quantitating the total extracted radioactivity will not suffice in most cases. If a method is to be used on both plant and animal commodities, it should be radiovalidated on a plant matrix, an animal tissue, and either milk or eggs. Matrices for which extraction is expected to be most difficult should be used. In the case of plants this would normally be a dry sample (e.g. straw, fodder) containing residues which have been on the plant for a relatively long period. Petitioners should provide a rationale for the samples used in the radiovalidation. If the data collection and enforcement methods have significantly different extraction steps, each method should be radiovalidated. Alternatively, analyses of several split samples bearing field weathered, nonradiolabeled residues showing similar results with the two methods may be submitted.

(ii) Methods utilizing only surface stripping are not acceptable except for commodities where extensive data have established that the TTRs are in fact only surface residues.

(iii) Certain components of the TTR may occur bound with naturally occurring plant constituents, and thus may not be recovered by extraction techniques that are satisfactory for the free components. Whenever there are indications of the formation of bound components which may not be recovered by the extracting solvent but are of toxicological concern, modifications should be made in the procedure that will free and recover the liberated components. One such modification would be the initial hydrolysis of the treated crop under acidic, basic, or enzymatic conditions to free the components. In some cases these bound components may also be recovered with polar solvents. These bound residues should not be confused with those fragmentary components which may be so tightly bound or incorporated into the plant's metabolic pool that they are not recoverable by any chemical means. Such components are of interest, but are not usually of toxicological concern. Refer also to the discussion on nonextractable residues in OPPTS 860.1300.

(4) **Determination of TTRs.** (i) The methods employed should measure the TTR found in the metabolism studies outlined in OPPTS 860.1300, Nature of the Residue. Often all components of toxicological concern will contain a common chemical moiety so that the method may be adapted to determine all compounds simultaneously. However, in some cases, it may be necessary to adapt separate extraction-cleanup procedures, or even another complete method, to measure the TTR or a significant component of the residue. In other cases, one or more components of the residue will be significantly more toxic than other components of the residue and will have to be determined separately.

(ii) In some cases the Agency will accept a tolerance enforcement method that measures only a portion (typically the parent compound) of the TTR in order to ease burdens on enforcement agencies and/or to harmonize with international maximum residue limits. This may be referred to as an indicator or marker compound. However, in order to have sufficient data for dietary risk assessment, a data collection method will normally still be needed to quantitate the TTR. Petitioners contemplating use of an indicator or marker compound in either enforcement or data collection methods are advised to contact the Agency about the acceptability of this approach.

(5) **Requirements for regulatory methods.** (i) One or more of the methods proposed in the petition must be acceptable to enforce the proposed tolerance. Where applicable, use of the FDA multidetection methodology outlined in paragraph (e)(6) of this guideline is strongly recommended. Also, the enforcement method should be as simple as possible to decrease the cost of monitoring for pesticide residues.

(ii) A method which may be valid for gathering residue data is not necessarily suitable for enforcement purposes. In general:

(A) An enforcement method should not require:

(*1*) The use of a sample of the untreated commodity as a blank.

(*2*) Exotic equipment or reagents (or reagents that are no longer manufactured).

(B) An enforcement method should be:

(*1*) Reasonably rapid in execution. In general, residue analytical methods for regulatory purposes should require one working day for completion. Methods taking longer than one working day will be considered acceptable on a case-by-case basis. Methods taking less than 24 hours (total time from initiation to completion of analysis) will be required for acutely toxic residues because of the possibility of enforcement action from accident or misuse situations.

(*2*) Sufficiently specific to measure and identify the residue in the presence of residues of other pesticides which could reasonably be expected to be present on the same commodity.

(*3*) Sufficiently sensitive in relation to the tolerance proposed.

(*4*) Practicable without the use of extremely hazardous or toxic reagents.

(iii) The Agency does not have a specific list of the types of analytical methods which are acceptable for enforcement of tolerances. Any procedure (e.g. GC, HPLC, MS, immunochemical) which meets the criteria described above would be acceptable. Methods based on cholinesterase inhibition are not regarded as suitable for enforcement purposes. Although methods based on paper or thin layer chromatography which visually measure the residue may not be adequately quantitative for enforcement purposes, they may be useful as confirmatory methods to help identify the residue.

(iv) Although certain gas and liquid chromatographic detection systems possess inherent specificity, methods based on these systems should usually be supplemented by a confirmatory method which is significantly different from the primary enforcement method. In general, confirmation by mass spectrometry is suitable. The specificity may also be enhanced by the use of special extraction-cleanup procedures, derivatization, or parallel and/or alternate columns. Provided that a specific confirmatory method is available, the Agency will not require that an interference study be conducted to show whether other pesticides registered on the same commodities interfere with determination of the pesticide of interest.

(v) The Agency accepts use of common moiety methods on a case-by-case basis. Toxicological differences among all metabolites of concern which can be determined by the method are taken into consideration when evaluating the suitability of a common moiety method. In those cases where a common moiety method is proposed as the primary enforcement method and other regulated pesticides produce the same common moiety, a confirmatory method specific for the residue of concern should be available to enforcement laboratories. This is especially critical in those instances where two pesticides generating a common moiety are registered on the same crop but have different tolerance levels.

(vi) The methods proposed for enforcement may be subjected to trials in Agency laboratories if the pesticide is new, if the analytical methods is new and unfamiliar, or if the commodity is known to be difficult to analyze. The burden of proof is on the petitioner, and should the method fail to perform as expected in these trials, the petitioner will be asked to resolve the difficulties. Also, the petitioner will be responsible for improving such a method and furnishing new residue data by the improved method. If the method performs satisfactorily and is acceptable as an en-

forcement method, it will be made available to interested parties by publication or reference in the PAM under paragraph (e)(7) of this guideline. Thus, a petition must include a copy of the analytical method which is not claimed to be, or stamped, confidential business information. Methods will be released to enforcement agencies prior to the establishment of a permanent tolerance if a FIFRA section 18 emergency exemption or a temporary tolerance is in effect. Prior to publication in the PAM under paragraph (e)(7) of this guideline, methods can be obtained directly from the Agency.

(6) **Independent laboratory method validations.** This paragraph describes performance of independent laboratory validation (ILV) trials for submission as part of the pesticide petition.

(i) ILV trials required to accompany petitions for tolerance. Results of ILV trials of new analytical methods are required for the parent pesticide, including metabolites of toxicological concern, and must accompany the following types of petitions:

(A) The first tolerance petition including temporary tolerance petitions for residues of a pesticide in a RAC or processed food/feed.

(B) Any new tolerance petition for residues of a pesticide with previously established tolerances if a new method is proposed for enforcement.

(C) Any new tolerance petition for residues of a pesticide with previously established tolerances if the previously approved enforcement method has been significantly modified to accommodate the new commodity. If the petitioner is uncertain whether a method change is significant, the Agency should be contacted.

(ii) Results of an ILV trial are usually not required for an enforcement method which the Agency deems superior to the currently accepted enforcement method. An ILV trial is also not normally required for confirmatory methods. However, at the discretion of the Agency, an ILV trial may be required for confirmatory methods on a case-by-case basis. One particular instance when the ILV trial is likely to be needed is for a confirmatory method associated with a compound whose primary enforcement method is a common moiety procedure which also detects other registered pesticides.

(iii) The laboratory personnel including the study director chosen to conduct the ILV trials must be unfamiliar with the method, both in its development and in its subsequent use in analyzing field samples. Provided this criterion is met and the same equipment, instruments, and supplies are not used, the laboratory chosen to conduct the ILV trials may be in the petitioner's organization. Other possibilities include laboratories at state enforcement agencies, at universities, or private laboratories. The pe-

titioner should apply the same criteria of quality in selecting a laboratory for ILV trials as would be done for any analytical work.

(iv) Requirements for ILV trial. (A) ILV trials must be conducted under FIFRA Good Laboratory Practice (GLP) standards as specified in 40 CFR part 160. A successful ILV trial will require adequate results for the TTR on at least one set of samples, and the laboratory conducting the ILV trial will be allowed to run up to three sets of samples using the method on a given commodity. A set of samples consists of two control samples, two control samples fortified at the proposed tolerance, and two control samples fortified at the LOQ. If the tolerance is proposed at the LOQ, the second fortification level should be twice the LOQ. At the discretion of the petitioner, one additional fortification at another level may be included in the set of samples. The method must be performed as written with no significant modifications. If additional commodities are analyzed by the same method, they will be considered to be separate ILV trials.

(B) The laboratory conducting the ILV trial may contact the developers or previous users of the method prior to running the first set of samples, but all communications must be logged and reported to the Agency. Under no circumstances should anyone from the petitioner, developer, or any previous users be allowed to visit the laboratory during the ILV trial to observe, offer help, or assist the chemists or technicians. If the first (or second) set is not successful, and the laboratory requires additional contact with the developers or other users of the method, all communications should be recorded. Any subsequent additions or modifications to the original method resulting in improved performance should be incorporated into the method write-up that is sent to the Agency for validation.

(C) If one method is to be used for several commodities, the ILV trial should be carried out on that commodity the petitioner has had the most difficulty analyzing. If the same method is used for both plant and animal commodities, separate ILVs should be run on both the most difficult plant and most difficult animal matrices. The rationale for selection of the commodity should be provided. If, after three sets of samples have been analyzed, the ILV trial has failed to produce adequate results, the petitioner must revise the method and perform a second confirmatory trial using a different laboratory.

(D) The results on one set of samples, after conducting no more than three sets, must be similar to those achieved by the petitioner to constitute a successful ILV trial. Recovery rates should be 70–120 percent and interference should be negligible compared to the proposed tolerance level.

(v) Information to be reported to the Agency. If the ILV trial is successful, the following should be submitted by the petitioner:

(A) Name, address, and telephone number of study director and other contact person for ILV laboratory.

(B) Description of the analytical method.

(C) All recovery and control values for all commodities that were obtained during all ILV trials.

(D) Representative chromatograms/spectra for each analyte in each matrix.

(E) Description of the instruments used and operating parameters.

(F) Description of any problems encountered and a written description of any changes or modifications that were made during the ILV.

(G) Any steps considered critical, i.e. steps where little variation is allowable or directions must be followed precisely.

(H) The number of worker-hours required to complete one set of samples.

(I) The number of calendar days required for one set of samples.

(J) Any contact between the independent laboratory and the method developers or others familiar with the method, including the reasons for the contact, any changes in the method that resulted, and the time of this communication with respect to the progress of the confirmatory trial (i.e. after the first set, during the second set, etc.).

(K) A statement of adherence to FIFRA GLP standards under 40 CFR 160.12.

(vi) The Agency will continue to conduct method validations. If the Agency determines that the petitioner has submitted results of a successful ILV trial by an independent laboratory, the method will be validated by Agency chemists.

(7) **Internal and procedural standards.** (i) The Agency accepts the addition of an internal standard to the final extract just prior to injection to serve as a calibration for retention times and/or peak heights/areas and to improve the precision of quantitation. However, the use of an internal standard throughout the entire procedure to correct for recoveries is not acceptable unless data are available on numerous samples of each matrix to show that the analyte and internal standard behave identically in each step (extraction, cleanup, etc.). For chromatographic methods, the peaks for the analyte and internal standard should elute close to one another, but be well resolved from each other. An example of an internal standard method which the Agency has accepted is the use of isotopically labeled dioxins for dioxin analysis by mass spectrometry (see paragraph (e)(2) of this guideline). As with any other reagent or reference standard used in

an enforcement method, the internal standard must be available to enforcement laboratories. If an internal standard is not commercially available, the petitioner must ensure a supply of the chemical to the Agency.

(ii) Procedural standards are considered to be standards which are generated by subjecting the reference standard to some or all of the sample preparation procedures specified in the method. The Agency will accept methods using procedural standards generated from a derivatization step under certain conditions. If a procedural standard is used, the petitioner should supply the Agency with not only the pesticide analytical standard, but also the derivatized standard. Availability of the derivatized standard would allow the enforcement laboratory to determine the efficiency of the standard preparation. If the derivatized standard is unstable or cannot be provided, the petitioner must provide data to demonstrate the efficiency and reproducibility of the procedure.

(d) **Data reporting format.** The following format is suggested for data reporting. However, other formats are also acceptable provided the information described in this paragraph is included.

(1) *Title/Cover page.* Title page and additional documentation requirements (such as requirements for data submission and procedures for claims of confidentiality of data) if relevant to the study report should precede the content of the study.

(2) *Table of contents.* The table of contents should provide page numbers for the essential elements of the study:

(3) *Body of report*—(i) *Introduction and summary.* (A) Scope (suitable matrices) and source of method (e.g. the PAM, company reports, etc.).

(B) Description of the principles of the analytical procedure, including identification of the chemical species determined and the limits of detection and quantitation.

(ii) *Materials and methods.* (A) Equipment—list and describe.

(B) Reagents and standards—list and describe source and preparation.

(C) Analytical procedure—detail in a stepwise fashion, with special emphasis on reagents or procedural steps requiring special precautions to avoid safety or health hazards.

(*1*) Preparation of sample.

(*2*) Extraction—demonstrate efficiency, if relevant (e.g. dry crop substrates, bound residues, etc.); radiovalidation data may be provided in a separate report at the discretion of the petitioner.

(*3*) Fortification, if applicable—i.e. during method validation runs.

(*4*) Clean-up.

(*5*) Derivatization (if any).

(D) Instrumentation—include information on:

(*1*) Description (e.g. make/model, type/specificity of detectors, columns (packing materials, size), carrier gases, etc.).

(*2*) Operating conditions (e.g., flow rates, temperatures, voltage, etc.).

(*3*) Calibration procedures.

(E) Interferences—describe tests.

(*1*) Sample matrices.

(*2*) Other pesticides.

(*3*) Solvents.

(*4*) Labware.

(F) Confirmatory techniques—describe.

(G) Time required for analysis (to carry a sample/set completely through the analytical procedure, including the determinative step).

(H) Modifications or potential problems, if any, in the analytical methods (detail circumstances and corrective action to be taken).

(I) Methods of calculation (describe in a stepwise fashion).

(*1*) Calibration factors.

(*2*) Analyte in sample.

(J) Other. Any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of residue analytical methodology and the means of calculating the residue results.

(iii) *Results and discussion.* Describe expected performance of method.

(A) Accuracy (expected mean and range of recoveries)—include, preferably in tabular format, the individual recovery values, average recoveries, and relative standard deviation thereof for each component of the TTR in each commodity tested during the petitioner's method validation.

(B) Precision.

(C) Limits of detection and quantitation (provide definition).

(D) Ruggedness testing, if performed.

(E) Limitations.

(iv) *Conclusions.* Discuss applicability of analytical procedure for measuring specific test compounds in various test substrates, ready availability of equipment, interferences, etc.

(4) *Certification.* Certification of authenticity by the study director (including signature, typed name, title, affiliation, address, telephone number, date).

(5) *Tables and figures*.

(6) *References.*

(7) *Appendices.* Include: Representative chromatograms, spectra, etc. (as applicable) and any relevant material not fitting in any of the other sections to this report.

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

(1) Association of Official Analytical Chemists (AOAC) International, Official Methods of Analysis, latest edition. Available from AOAC International, 2200 Wilson Boulevard, Arlington, VA 22201.

(2) Environmental Protection Agency, EPA Test Methods for Evaluating Solid Waste (SW–846), Method 8280 and EPA Test Methods for Organic Chemical Analysis, Method 613.

(3) Environmental Protection Agency, PR Notice 96–1, Tolerance Enforcement Methods—Independent Laboratory Validation by Petitioner, Feb. 7, 1996.

(4) FEDERAL REGISTER 58:50888–50893, Sept. 29, 1993.

(5) Horwitz, W. et al, Quality Assurance in the Analysis of Foods for Trace Constituents, JAOAC, Vol. 63, No. 6, pp. 1344–1354 (1980).

(6) Pesticide Analytical Manual. Food and Drug Administration. Volume I. (1994). Available from the National Technical Information Service, Springfield, VA.

(7) Pesticide Analytical Manual. Food and Drug Administration. Volume II. (1994). Available from the National Technical Information Service, Springfield, VA.