United States Environmental Protection Agency Prevention, Pesticides and Toxic Substances (7101) EPA 712-C-96-169 August 1996

Residue Chemistry Test Guidelines

OPPTS 860.1000

Background

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 860.1000 Background

(a) Scope--(1) Applicability. This guideline is intended to meet testing requirements of both the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) (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 materials

used in developing this harmonized OPPTS test guideline are OPP test 170-1 Scope of Data Requirements, OPP 171-1 List of Requirements, OPP 171-14, Special Considerations for Temporary Tolerance Petitions, and OPP 171-5 Presentation of Residue Data (Pesticide Assessment Guidelines, Subdivision O: Residue Chemistry, EPA Report 540/9-82-023, October 1982).

(b) Purpose and scope of data requirements--(1) General. (i) This guideline provides general information and overall guidance for the 860 series on residue chemistry and registrants should use it in conjunction with the other listed guidelines to assure compliance with registration requirements. Topics addressed in this guideline include: Purpose and scope of data requirements, regulatory authority, minor change in use pattern, food use/nonfood use determinations, tobacco use, aquatic uses, special considerations for temporary tolerance petitions, data requirements for temporary tolerances, presentation of residue data, guidance on submittal of raw data, and references. Also included (under paragraph (m) of this guideline) is a table ``Raw Agricultural and Processed Commodities and Livestock Feeds Derived from Field Crops."

(ii) Sections of this series describe the residue chemistry data requirements specified by 40 CFR part 158, the Federal Food, Drug and Cosmetic Act (FFDCA), and some other information needed for pesticide uses that may result in residues in food, feed, or tobacco. Residue chemistry data are used by the Agency to estimate the exposure of the general population to pesticide residues in food, and for setting and enforcing tolerances for pesticide residues in food or feed. (iii) Information on the chemical identity and composition of the pesticide product, the amounts, frequency and time of pesticide application, and results of test on the amount of residues remaining on or in the treated food or feed, are needed to support a finding as to the magnitude and identity of the residues which result in food or animal feed as a consequence of a proposed pesticide usage. (iv) Residue chemistry data are also needed to support the adequacy of one or more methods for the enforcement of the tolerance, and to support practicable methods for removing residues that exceed any proposed tolerance.

(2) Petitions for tolerance. Residue chemistry data for a new use of a pesticide are generally submitted to the Agency in a petition for tolerance as required under section 408 or 409 of the FFDCA. The format, procedures and fees associated with petitions for tolerance are included in sections 408 and 409 of the FFDCA, and 40 CFR 180.1 to 180.35.

(c) Regulatory authority. The Agency regulates pesticides under two acts, FFDCA and FIFRA. The FFDCA gives the Agency the authority to set legally enforceable limits, or tolerances, for pesticides in foods. The Agency sets tolerances for pesticide residues remaining in raw agricultural commodities (RACs) under section 408 of the FFDCA. The Agency sets food additive tolerances under section 409 of the FFDCA for pesticide residues which concentrate in processed foods above raw food tolerances, or which are the

result of pesticide application during or after food processing. In some cases maximum residue limits are established under section 701 of the FFDCA for residues in processed commodities. Under FIFRA, all pesticides must be registered with the Agency before they may be sold or distributed in commerce. FIFRA sets an overall risk/benefit standard for pesticide registration, requiring that pesticides perform their intended function, when used according to labeling directions, without posing unreasonable risks of adverse effects to human health or the environment. (d) Minor changes in use pattern. (1) If a minor change in the use pattern or formulation of a currently registered pesticide is requested, the registrant may have to submit additional residue data to demonstrate that the change will not result in residues exceeding the established tolerance. Examples of changes in pesticide use patterns that are likely to require residue studies, and possibly another petition for a new tolerance, include:
(i) Significant changes in preharvest interval and/or in postharvest treatment.
(ii) Extension of use patterns to include low volume or ultralow volume (ULV) aerial as well as ground application. (iii) Addition of a sticker or extender to the formulation. (iv) Conversion to a slow-release formulation. (v) Use in additional climatic regions. (vi) An increase in the application rate. (vii) An increase in the number of applications allowed. (2) Examples of minor changes that may not require residue studies include:
(i) A change in surfactant concentration. (ii) Substitution of a new but similar surfactant. (iii) Substitution of one clay diluent for another.
(iv) Exceptions must be made individually based on a thorough knowledge of the chemistry of the ingredients involved. (3) Residue data in support of a changed use pattern are normally submitted to the Agency under FIFRA in a form described in 40 CFR part 162.
(e) Food use/nonfood use determinations--(1) Definitions. The term ``food'' is defined in section 201(f) of the FFDCA as articles used for food or drink for man or other animals, chewing gum, and articles used for components of any such articles. If a pesticide use is likely to result in residues on food, the use is a food use, a petition for tolerance or exemption is required, and appropriate residue chemistry considerations apply. Nonfood uses are those uses that are not likely to yield residues in food. Uses that could result in residues in meat, milk, poultry, or eggs are also considered to be food uses. (2) Food use/nonfood use determination data requirements. (i) In some cases residue chemistry data are needed to determine whether a proposed use is a food use or a nonfood use. The general criteria for food use/nonfood use determinations is that if residues could occur in foods or feed, the use is a food use and a petition for tolerance/ exemption from tolerance is required. In some cases this determination can be made based on the nature of the site to which the pesticide is to be applied. Thus application to land other than cropland is

considered a nonfood use. In other cases the distinction is not as clear. For example, baiting with a rodenticide around the borders of cropland or in a tamper-resistant bait box within cropland would be considered a nonfood use, but applying the bait directly to the crop would be considered a food use.

(ii) For the following types of uses, the food use/nonfood use determination will be based on the results of the data described in each section. Registration for these types of uses will not be granted until the necessary data have been submitted to the Agency and found acceptable.

(A) Seed treatments. (1) This includes cases where seed is treated either by the seed company (and dyed according to 40 CFR 153.155) or by the farmer (planting box or hopper treatments). (2) In order for a seed treatment to be considered a nonfood use, data from a radiotracer study must be available showing no uptake of residues (radioactivity) from treated seed into the aerial portion of the growing crop.

(3) If residues occur in the aerial portion of the plant, or if there is no data available to make this determination, seed treatments are considered to be food uses requiring tolerances. (B) Crops grown for seed only. (1) These crops may qualify as nonfood uses provided there is no likelihood of residues in crops grown from the harvested seeds and the seeds or other parts of the treated crop are not diverted to food/feed use. Factors affecting this include the level of residues on the harvested seed, the half-life of seed residues, the weight of the seed in relation to that of the subsequent crop, and the amount of residue uptake from the seed into the aerial portion of the crop. More details are provided in paragraphs (e)(3) and (e)(4) of this guideline.

(2) On the other hand, uses on crops where the seed itself is a major RAC (such as corn, sorghum, soybeans, small grains, and sunflowers) are considered to be food uses. In these cases, seeds from treated crops could not be distinguished from untreated crops and could be diverted to human and animal consumption. (3) With the exception of certain FIFRA section 24(c) registrations (also referred to as special local needs (SLNs)), as discussed in paragraph (e)(2) of this guideline, alfalfa and clover grown for seed cannot be considered a nonfood use because of the economic importance of alfalfa and clover hay. Subsequent cuttings for hay could be taken regardless of label restrictions. Also, because of the increasing importance of alfalfa sprouts as a human food item, use on alfalfa grown for seed only cannot be considered a nonfood use. (4) Desiccation uses for clover grown for seed will be considered nonfood uses because the desiccation renders the hay unfit for consumption by livestock.

(C) Fallow land. Use of a pesticide on fallow land requires data indicating whether residues persist in soil long enough for uptake by crops. Fallow land uses must include a time limitation on planting to food/feed crops or tobacco. Twelve months is the longest time interval deemed practical for a fallow land use restriction. If residues persist in soil and are taken up

by food/feed crops for the length of the time of planting limitation, or 12 months (whichever is shorter), a petition for tolerance for all crops which could be planted on the fallow land will be required. Additonal guidance is provided in OPPTS 860.1850 and 860.1900.

(D) Nonbearing crop uses. Nonbearing crops are perennial crops that will not produce a harvestable RAC during the season of application. Application of a pesticide to a nonbearing food or feed crop will be considered a nonfood use only if data are available to demonstrate that no detectable residues occur in the crop at the first harvest. However, provided the label contains a restriction against harvesting food/feed within 1 year of application, the Agency will consider such uses to be nonfood unless the pesticide is known to be persistent and systemic as shown, for example, by rotational crop data. If residues are detected in the crop at first harvest, a petition for tolerance with the full range of residue chemistry data requirements will be needed before a tolerance and registration will be granted. (3) Additional considerations. (i) One area in which numerous questions have arisen over the years is whether uses of pesticides on crops grown for seed are nonfood uses, which do not require tolerances. The Agency's concern has been that the crop may be diverted to food use before seeds are produced or that the seeds themselves may be used for food or feed, regardless of label statements prohibiting such practices. (ii) In recent years the Agency has accepted some uses on crops grown for seed as nonfood uses. If a State is willing to provide assurance that seed crops will not be diverted to food or feed and information on acreage and cultural practices is provided, the Agency will consider pesticidal treatment of crops grown for seed as a nonfood use under FIFRA section 24(c) registrations. The information on cultural practices should emphasize those practices which distinguish the seed crop from the corresponding food crop and which would render the seed crops commercially unviable for food. Examples of such practices might be smaller crop spacing preventing adequate root formation and planting in a different season to encourage bolting versus head formation. Information would also be needed on possible residues in followup crops grown from the harvested seeds as described in paragraph (e)(4)(i)(B) of this guideline.

(iii) The Agency will consider these uses on a case-by-case basis for FIFRA section 24(c) registrations once the assurances and cultural practice information outlining a rationale as to why these are nonfood uses have been submitted by a State. Most cole, leafy vegetable, and root crops have a good chance of obtaining pesticidal treatment of their seed crops on a nonfood basis. Crops where the seeds themselves are major RACs, such as grains, beans, and peas, have very little chance of such registrations on a nonfood basis. Alfalfa, clover, and grass grown for seed are generally not considered nonfood uses since they can be cut for hay prior to going to seed. However, the Agency has accepted use on alfalfa grown for seed in certain states, including Washington State, based on rules issued by the

States governing the use of the seeds, seed screenings, and other crop parts. Recently, the Washington State rule was expanded to cover more than 30 small-seeded leafy and root vegetables.
(4) Seed use/nonfood use special local needs. (i) Seed use may refer to direct application of a pesticide to seeds before planting (seed treatments) or application in the field to crops grown for seed. This section clarifies the type of information that the Agency needs so that a FIFRA section 24(c) or SLN for seed use may be deemed to be nonfood. If the Agency has a legitimate concern about carryover to subsequent crops, residue information will be needed. (A) Seed treatments. Seed treatment uses can be considered nonfood only if a radiotracer study shows no uptake of radioactivity into the aerial portion of the crop (or into the underground portion of root crops). Our experience with such studies is that it is quite unlikely a seed treatment will be considered a nonfood use. In most cases a tolerance is established at the quantitation limit of the analytical method on the crop grown from the treated seeds. (B) Applications to crops grown for seed. Applications to crops grown for seed can be considered nonfood uses if the following two conditions are met:
(1) Subsequent to treatment no parts of the crop will be diverted to use as human food or livestock feed. (2) There is no likelihood of residues in crops grown from the harvested seed.
(3) These conditions are discussed in more detail under paragraphs (e)(4)(ii) and (e)(4)(iii) of this guideline. (ii) (A) In some instances the first condition may be met by the timing of the application. In other words, the condition is met if the pesticide is applied to the seed crop at a point when it is no longer fit for consumption. An example would be use of a desiccant on carrots or radishes near the time of seed harvest. At this time the roots would no longer be desirable as a food.
(B) In those cases where the application timing does not satisfy the first condition, two other possible means of meeting that condition exist:
(1) The State in which the registration is sought provides assurance through some regulatory process that the seed crop will not be diverted to food or feed. This assurance must include all crop parts that could be consumed by humans or livestock. Crops of special concern are alfalfa, clover, and grass, which may be cut for hay. The first example of a State using this procedure was Washington State for registration of pesticides on alfalfa grown for seed. (2) Cultural practices information is submitted showing how the seed crop may be distinguished from the corresponding food crop and how the seed crop is commercially unviable as a food crop. Examples of such cultural practices might be smaller crop spacing preventing adequate root formation or planting in a different season to encourage bolting versus head formation.
(C) Some general statements can be made at a crop grouping level with regard to the chances of the first condition being met. Most cole, leafy vegetable, and root crops grown for seed have a good chance of meeting this

requirement. On the other hand, crops where the seeds themselves are major RACs such as grains, beans, and peas have very little chance of nonfood registrations. Cucurbits and fruiting vegetables are probably not eligible for nonfood uses since the fruit is still edible at the stage when the seeds have formed. (iii) The second condition to be met for a nonfood use will be addressed:

(A) In many cases this condition may be met without actual residue data on the harvested seed or the crop grown from that seed. The registrant and/or State should consider data or information on the following factors when calculating a theoretical residue in the crop grown from the harvested seeds.

(1) Weight of seed.

(2) Weight of edible portion of following crop. (3) Total weight of following crop. (4) The weights could be expressed in terms of an individual seed/plant or on a per acre basis. In either case the figures would allow an estimate of the dilution of residues due to growth of the plant.

(5) Tolerances on other crops with similar rate and preharvest interval.

(6) Information as to whether the seeds are directly exposed to the pesticide spray. (The above two factors can be used to estimate maximum likely residues on the harvested seeds.) (7) Seed treatment data for the pesticide on other crops. (8) Degree to which the pesticide translocates (how systemic?). (9) Half-life of the pesticide on other crops. (The above three factors can be used to estimate how much the pesticide might move from seed to the growing crop.)

(B) [Reserved]

(iv) If a calculation using reasonable assumptions and taking into account the above factors indicates residues in the RAC grown from the harvested seeds will be well below (for example, 1 order of magnitude) the detection limit of the analytical method, condition two would be met. On the other hand, if the calculation shows residues close to or above the detection limit, actual residue data on the harvested seeds and/or following crop will be required to show that the use can be considered nonfood.

(f) Tobacco uses. Use of pesticides on tobacco does not require a tolerance or an exemption from a tolerance. Nonetheless, data are needed to assess the exposure of humans to residues on tobacco. The following residue chemistry data for tobacco uses should be submitted to the Agency using the procedures for submitting data under FIFRA referred to above.

(1) Nature of the residue study. A tobacco metabolism study is required. This study is similar to plant metabolism studies required in support of tolerances on RACs and discussed in OPPTS 860.1300. The commodity of interest is green tobacco leaves. If total toxic residues (parent plus metabolites of potential risk concern, as determined by the Agency) in green tobacco are <ls-thn-eq> 0.1 ppm, no further residue chemistry data are required. (If the Nature of the Residue in plants is considered

adequately understood based on plant metabolism studies accepted by the Agency for uses on food crops, the registrant may choose not to perform a tobacco metabolism study, but may move immediately to generation of field trial data. However, it should be noted that if total toxic residues <ls-thn-eq>0.1 ppm are found in a tobacco metabolism study, field trial data would not be required.) (2) Field Trials. (A) If metabolism data show that the total toxic residue (TTR) is <ls-thn-eq>0.1 ppm in green tobacco, a validated analytical method (see OPPTS 860.1340 for basic requirements) and field trial data are required. Field trials should be conducted, as with food crops, using the maximum application rate and number of applications, minimum intervals between applications, and minimum preharvest interval. Samples of green tobacco should be analyzed for the total toxic residue identified in the metabolism study.
(B) Adequate geographical representation is required. OPPTS 860.1500 indicates that three field trials are required (which should all be performed in Region 2). Other applicable requirements in that guideline such as number of samples per site should also be met. (C) If the maximum residue in all individual composite samples of green tobacco is <ls-thn-eq>0.1 ppm, no additional residue chemistry data are required. If the maximum residue in any individual composite sample of green tobacco is <ls-thn-eq>0.1 ppm, residues in samples of cured tobacco should be determined (three locations may be the same as those for green tobacco). If the maximum residue in all individual composite samples of cured tobacco is <ls-thn-eq>0.1 ppm, no additional residue chemistry data are required.
(3) Pyrolysis study. If the maximum residue in any individual composite sample of cured tobacco is >0.1 ppm, a pyrolysis study is required. Pyrolysis products resulting from the total toxic residue must be identified/characterized in a manner similar to that required for plant metabolism studies. Information from the pyrolysis study will be presented to the Toxicology Branches and/or the Health Effects Division (HED) Metabolism Committee for determination of their human health significance.
(g) Aquatic uses. When a pesticide is applied directly to water, data on potential residues in water, fish, shellfish, and irrigated crops will be required. Tolerances are not established for potable water under FFDCA, thus data on residues in water are submitted under FIFRA. Residue limits for potable water will be established under the auspices of the Safe Water Drinking Act (SWDA) when appropriate. Tolerances for fish, shellfish, and/or irrigated crops should be submitted in the form of a petition for tolerance or exemption from tolerance under the FFDCA. (h) Special considerations for temporary tolerance petitions. (1) A temporary tolerance may be established in conjunction with an Experimental Use Permit (EUP). A petition for a temporary tolerance should specify the amount of pesticide which will be used under the conditions of the experimental permit, the crop acreage which will be treated, and the geographical areas where the treatments will be made. (2) The chemistry data requirements for permanent

tolerances will in general apply to petitions for temporary tolerances except that the metabolism and residue data need not be extensive. Whether the latter exception is to be made will depend on the toxicity of the pesticide or possible degradation products and/or metabolites, the amount of acreage to be treated, the importance of the food or feed commodity, and similar considerations. It is assumed that data will be obtained while the EUP is in effect and will be made available to the Registration Division of OPP.

(i) Data requirements for temporary tolerances. (1) Temporary tolerances are established to cover residues of pesticides that result from EUPs. These permits are used to gather additional data on efficacy, phytotoxicity, and pesticide residues. The locations, acreages, amounts of pesticide to be applied, and the names of cooperators must be specified. In addition, these EUPs allow use of the pesticide on only a very small percentage of the total national acreage of a crop. For these reasons, the data requirements for a temporary tolerance are not as stringent as those for a permanent tolerance. This guideline provides more details as to what is meant by the phrase ``need not be as extensive".

(2) With regard to metabolism studies, characterization and/or identification of the residue need not be as thorough as required for permanent tolerances. If the parent pesticide is the predominant residue in that portion of the residue which has been identified, it is usually acceptable to regulate only the parent compound. However, additional characterization and/or identification of the residue will be required for permanent tolerances. It may also be acceptable to defer regulation of major metabolites (e.g. those comprising more than 10 percent of the total residue) to the permanent tolerance stage. Such decisions will be made via the temporary tolerance review and consultation with the HED Metabolism Committee.

(3) For plant metabolism, the usual requirement for studies on three diverse crops need not be met for temporary tolerance petitions. In those instances where no detectable residues are found on feed items (assuming detection limits for feed items are <ls-thn-eq>0.1 ppm), livestock metabolism studies may be delayed until the permanent tolerance stage. In addition, it may be possible to delay poultry metabolism studies to the permanent tolerance stage when low residues are found in feed items and a ruminant metabolism or feeding study indicates there is little transfer of residues to tissues or milk. (4) Petitioners will be required to submit enforcement methods for temporary tolerances. The methods must be accompanied by independent laboratory validations as directed by PR Notice 96-1 (see paragraph (n)(5) of this guideline). EPA laboratory validations will also be initiated as part of the temporary tolerance process. However, if no other data deficiencies exist, recommendation for a temporary tolerance should not be held up pending the completion of EPA's method validation. If the EPA method trial fails for reasons of enforcement

practicality (e.g., availability of glassware) but shows the method is valid for collection of residue data, approval of temporary tolerances may proceed, but the petitioner should be informed to correct the problem prior to establishing permanent tolerances. On the other hand, if a method fails and is not valid for the collection of residue data (due to low or erratic recoveries, for example), the validity of the residue data needs to be determined. Any future tolerances (temporary or permanent, including extensions of temporary tolerances about to expire) depending on that method may not receive a favorable recommendation. (5) Data showing whether the FDA multiresidue procedures recover a pesticide may be delayed until submission of a permanent tolerance petition.

(6) The number and/or geographic representation of crop field trials need not be as complete for temporary tolerance petitions. Translation of residue data from similar crops may also be more liberal than that employed in review of permanent tolerances. For example, if use patterns are essentially the same, field trials on apples may be used to supplement pear data for establishing a temporary tolerance on the latter. Storage stability data can also be translated from different crop groups at this stage.

(7) Since temporary food and feed additive tolerances may be established, processing studies are normally required to determine whether residues concentrate in the processed commodities listed in Table 1 under paragraph (m) of this guideline. In some instances it may be possible to waive the processing study if no detectable residues are found in the RAC following application of exaggerated rates (see discussion of processed food/feed under OPPTS 860.1520). (8) Certain label restrictions may be considered practical for an EUP that would never be considered such for full registration. For example, if an extremely small acreage of grapes were to be treated, it might be practical to restrict the grapes to the fresh market only. Another case may be restricting the feeding of wheat straw to livestock. If such restrictions can be deemed practical for a particular EUP, data on certain RACs or processed commodities (e.g., grape juice, grape pomace, and raisins in the first case cited above) may be waived for the temporary tolerance approval. However, the petitioner should be informed that data are needed to remove such restrictions for a permanent tolerance.

(9) With regard to product chemistry data, information on beginning materials, the manufacturing process, and some analyses of the technical grade active ingredient are required. This may be provided on batches produced by a pilot plant. Data on physical/chemical properties may be deferred to permanent tolerance petitions. (10) The Agency normally does not object to the extension of EUPs and the accompanying temporary tolerances for a year or two, provided a significant expansion of acreage has not been requested. (j) Presentation of residue data. (1) Individual analyses, not average results, should be reported. The data should include

blank values and uncorrected values for the treated samples. If applicable, it should be indicated how corrections have been made for blanks and recoveries. When analytical methods that rely upon retention time for chemical identification (e.g. gas-liquid chromatography (GLC) and high performance liquid chromatography (HPLC)) are used, corrections should not be made for blanks due to discrete peaks (at the retention time of the pesticide sought) in the chromatograms of untreated controls. (2) It is preferable to summarize the data in tables showing crop, residue found, dosage, intervals between treatments and interval from final treatment to harvest (PHI), number of applications made, and formulation used. However, the tabulated data should be keyed for ready reference to the raw data and sample history sheets. The sample history sheets should note rainfall, sample treatment (washed, brushed, trimmed, etc.), sample collection and analysis dates, storage conditions, and other factors which might affect the residue levels. (3) Standard curves, optical absorbance readings, or copies of appropriately labeled chromatograms should be included in the raw data. It is desirable that at least some of the chromatograms show the analyst's sample dilution factors, sample equivalent injected, and the way in which peaks were quantitated. Photographs of thin-layer chromatography plates, paper chromatograms, or radioautographs of plants treated with labeled pesticides should be furnished when such evidence is necessary for the evaluation of the data. More detail on raw data appears in paragraphs (k) and (l) of this guideline. (4) A statistical treatment of data may be used to express the precision and accuracy of the analytical results when sufficient data make such treatment valid. The null hypothesis technique is useful in calculating the confidence level at which a set of values from control samples do not differ significantly from a set of values from treated samples. A graphical representation of residues versus time (decline or dissipation rate curves) is also desirable. This is usually plotted on semilog coordinates with time (days after treatment) as the linear function.

(k) Guidance on submittal of raw data. (1) The distinction between summary tables and raw data is very important. According to the Good Laboratory Practice (GLP) standards under 40 CFR 160.3, raw data are defined as

> * * * any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. * * * ``Raw data'' may include, but is not limited to, photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

(2) A study report is designed to provide specific information in order to fulfill an Agency requirement. It must include a description of the methods

and materials used, the results of the experiment, a discussion of those results, and a conclusion should be made regarding the objective of the study. Calculations or statistical evaluations should be clearly presented and sufficient raw data must be submitted with the report so that the Agency can completely understand how the study was conducted and how the results were derived. Registrants may also want to consider submitting additional information with some studies if they feel it would enhance the review process. (3) There is a considerable amount of information which is not necessarily considered to be raw data, but which can be crucial for evaluation of a study. Therefore the Agency has decided to include not only requirements for raw data, but also any critical information which is necessary for evaluation of a study and may not be discussed in other guidelines.

(4) The first portion of paragraph (l) of this guideline describes general raw data requirements which apply to virtually all residue chemistry studies. Individual types of studies (i.e. metabolism, analytical method, field trials, etc.) are discussed. Examples are provided whenever specific problems have been noted or special circumstances may exist. Original raw data should not be sent; copies are sufficient. Transcription of data may be adequate for some situations, particularly if the original data are illegible. (5) Other considerations. (i) When preparing reports and organizing the raw data associated with the final report the registrant should ensure that information is provided in a manner which is complete, legible, and logical. The data should be well-organized so that the Agency can easily find the desired information. Each sample value listed in a summary table should be linked to a field report, chromatogram, and quantitative data worksheet. Sample storage information and dates should be readily available so the Agency does not need to look at many different places in a report to find the dates of application, harvest, shipping, sample transfer, extraction, and analysis.

(ii) The registrant may want to consider tabulating much of the data into a spreadsheet or other appropriate format. For example, residue data could be organized in a spreadsheet which includes all information necessary for calculation of the residues such as sample weight, volume of final extract, peak height/area, etc. so the Agency can verify the reported results. It would not be necessary to submit each and every calculation sheet if the results were tabulated in such a manner.

(iii) Chromatograms should be completely labeled. A time scale should be on the chromatogram along with the complete sample identification. The retention times of the peaks of interest should also be noted on the chromatogram.

(iv) Data are often submitted which are illegible after copying once or twice and cannot be reproduced readily on microfiche. If the original raw data will not be legible after copying, the data should be transcribed, a statement should be made that the data have been transcribed (or included

in a worksheet or spreadsheet), and the transcriptions should be submitted. Raw data are not useful to the Agency if the data cannot be understood. Thin-layer chromatograms (from metabolism studies) are particularly difficult to read when photocopied. Extra attention should be paid to reproductions of these data. (v) A complete table of contents listing any data appended should be in the report. If a checklist of raw data is appropriate, the registrant may want to consider including such a list. (vi) A summary of data from associated studies (e.g. storage stability, bridging formulations, etc.) would be helpful, along with a specific reference, including Master Record Identification (MRID) number. The Agency frequently spends a considerable amount of time tracking down such information. While this is not raw data, inclusion of such a table could substantially reduce review time. (l) Requirements for residue chemistry studies--(1) General. Most residue chemistry studies have several common elements: The pesticide is applied to a crop or animal, samples are taken from crops or animals, samples are stored prior to analysis, and the samples are subjected to an analysis. The following list of raw data requirements will apply to most types of residue chemistry studies. (i) Field notes and/or reports on application, harvest, and plot maintenance. (ii) Calibration of application equipment (for confirmation of application rate).

(iii) A specific description (which may be in the field notes) as to how and where (within the plot) the sample was taken. What was done to ensure that a sample is representative of the test plot? (iv) All dates (not just intervals) associated with the study: Planting (if applicable), application, harvest, shipping, processing, sample preparation, extraction, and analysis. Tabulation in a single table along with the calculated intervals would be helpful. (v) Information on reference substance characterization as required by GLP standards.

(vi) Techniques used for preparation of the subsamples used for analysis.

(vii) If automated data calculation methods are used, some discussion/examples on the calculation techniques, such as the spreadsheet formulas, calculation parameters from a commercial system, etc.

(viii) When reports reference registrant/laboratory standard operating procedures (SOPs) and standard methods. The registrant must ensure that descriptions of any critical referenced procedures must be available to the Agency at the time of the review. If these SOPs have been previously submitted to the Agency, the registrant may reference the documents by the MRID number or accession number. (ix) Data from analyses aborted due to instrument failure or other circumstances should not be included. GLP regulations require that such data be kept, but it need not be submitted with the final report. (2) Requirements for plant and animal metabolism studies. (i) The purpose of these studies is to define the chemical nature of the pesticide residue in plants and animals. Because all other residue chemistry studies depend on the residue definition, these are the most

important residue chemistry studies considered. Review of metabolism studies is time consuming because they are so closely scrutinized. Generally more raw data are required for metabolism studies than any other type of study. It can be discerned from the list below that all the raw data associated with any analysis must be submitted. Ancillary raw data such as freezer temperature logs, standard preparation records, and instrument maintenance logs need not be submitted. (ii) It must be emphasized that raw data must be submitted. Corrections for losses should not be made. Requirements for raw data are outlined in the following list. These items should be considered in addition to the general list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline.

(A) All quantitative data which are used to assess the amount of radioactivity in all samples and extracts. This would include, but is not limited to, specific activity of the radiolabeled pesticide and quantitative details of its dilution (how much labeled and how much not labeled) in the final formulation, counting data (in disintegrations per minute), sample weights, counting efficiencies, and other appropriate measurements so that the concentration of radioactivity (test chemical) may be verified. Data should be provided for control and treated samples. Background data for solvents must be included as well. (B) All chromatograms, mass spectra, NMR spectra, infrared spectra, UV/vis spectra, and any other data used to make a determination of the characterization and identification of degradation products. This would include chromatograms or spectra which demonstrate negative results (i.e. proposed metabolites are not found). (C) Quantitative data associated with chromatograms, autoradiograms, spectra, etc., so that quantitative assessment can be made of metabolite identification of degradation products. This would include the amount of radioactivity (in disintegration per minute) used in the analysis (e.g. applied to the TLC plate) and the amount recovered. A complete set of sample calculations should be included with formulas and variables defined in generally understood terms, using data from the submitted report.

(D) Chromatograms/spectra of standards and controls. (E) A flow chart is a useful format for presenting data on distintegrations per minute for extractions. (F) Information on sample and extract storage including conditions, temperatures, intervals, etc. (G) The route of exposure to the animals should be specifically described. This is particularly crucial for dermal treatments; location on the animal's body should be described. (H) Vital statistics of the test animals throughout the study including body weights, egg or milk production, and feed consumption. Animal housing should be described as well. (3) Requirements for residue analytical methods studies. Once the pesticide residues of concern have been determined from the metabolism studies, analytical methods must be developed so the residues of concern in the commodities of interest can be quantified. There are three types of studies which are submitted under this

category: Residue methods specific for the residues of concern which may be used for collection of residue data and enforcement of pesticide tolerances, independent laboratory validation of the tolerance enforcement method, and testing of the pesticide and its residues of concern through the FDA multiresidue protocols. Requirements for raw data are outlined in the following list. These items should be considered in addition to the general list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline.

(i) All validation data (recoveries) associated with all methods submitted for all commodities of interest. This would include not only final results, but also sample weights, extraction volumes, final volume of extracts, peak heights/areas, injection volumes, and any other data which would allow the Agency to reproduce the calculations. These data can easily be summarized in a table, and may be reported along with analytical data for treated samples.

(ii) Chromatograms from all commodities of interest at all spiking levels, including the claimed limit of quantitation (LOQ). A minimum of 10 chromatograms is suggested for each commodity including both split and control samples, particularly in those instances where tolerances are proposed at or near the limit of quantitation. There is a need for more control and method blank chromatograms at this time than is generally submitted so the Agency can assess the reported limits of detection and quantitation. (If the method has been previously submitted to the Agency for other commodities, it is not necessary to resubmit data, and it may be referenced by MRID number). (iii) If multiple commodities are tested by the independent laboratory, data (including chromatograms) for all tested commodities should be submitted.

(iv) Notes on all technical communications between the study sponsor and the independent laboratory validating the method. This could be summarized by the analysis in a report rather than being notes from a laboratory notebook.

(v) Analyst notes on the difficulties of the method and modifications made to facilitate method implementation. (vi) All appropriate manufacturer and lot numbers for chromatography columns, chemicals, and equipment. (4) Requirements for storage stability. Most samples collected for later analysis in conjunction with a metabolism or magnitude of residue study are stored for an extended period (more than 1 month) prior to analysis. Therefore registrants must assure the Agency that the residues are stable in the commodity during the storage period under the conditions actually used for storage of field samples or demonstrate the degree of loss of residues during storage. Storage stability studies are designed to provide this information. Requirements for raw data are outlined in the following list. These items should be considered in addition to the general list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline. Separate guidance on storage stability studies is available under OPPTS

guideline 860.1380. (i) Storage intervals, locations, temperatures, containers. (ii) Preparation of samples prior to placement into storage. The report should state whether the samples were stored intact or homogenized.
(iii) Dates of spiking, extraction, and analysis. (iv) If samples with field-weathered residues are used, a summary of the field trials used to generate the samples (refer to the section on crop field trials). Registrants may choose to reference this information (MRID number) if it has been submitted to the Agency in a separate report.
(v) All quantitative data so that the Agency can reproduce the calculations performed by the registrant. This would include, but not necessarily be limited to, sample weights, extraction volumes, aliquot volumes/weights, injection volumes, final extract volumes, and peak heights/areas. This may be summarized in a table or spreadsheet. (vi) Representative chromatograms of control, spiked, and treated (if applicable) samples, including those used to demonstrate storage stability in metabolism studies. (5) Requirements for magnitude of the residue studies. (i) Several types of residue chemistry studies are used to obtain a quantitative assessment of the residues of concern in/on commodities consumed by humans and/or livestock. Table 1, under paragraph (m) of this guideline, identifies all significant food commodities and feed stuffs, both raw and processed, for which residue data are collected and tolerances are set. Table 1, titled ``Raw Agricultural and Processed Commodities and Livestock Feeds Derived from Field Crops," was Table II in the earlier OPP guidelines. In addition, the table provides the following information for feed stuffs: A maximum percent of the diet on a dry weight or as-fed basis for beef and dairy cattle, poultry, and swine, and guidance on the acceptability of label restriction prohibiting acommodity's use as a livestock feed. The Agency recognizes all plant commodities can be, and are, fed to livestock. However, the Agency requires livestock metabolism and feeding studies, and residue data only on those commodities considered to be ``significant."
(ii) Most raw data requirements are common to each type of magnitude-of-the-residue study. Therefore general requirements are described followed by requirements which would be unique to each type of study. These items should be considered in addition to the general list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline.
(A) Specific information on sample storage including conditions, sample form (intact or homogenized), and sample container. Often a range of temperatures is provided, which account for some temperature spikes, but a statement as to the sample condition throughout the storage period is generally not provided. For example a power outage may have driven the temperature (of the freezer) up to 5 deg.C from -20 deg.C, which is duly reported, but the Agency does not know how long the samples were stored at 5 deg.C or whether they remained frozen. Magnitude of residue study reports should detail how samples are handled and stored prior to receipt by the

lab. In particular, the interval between sample harvest and refrigeration of the sample (or placing it on Dry Ice) should also be reported.
(B) A sufficiently wide range of chromatograms must be submitted so that the Agency can make an assessment of the reproducibility of the method, potential interferences, variations in signal-to-noise ratios, and so on. If a study consists of 10 samples or fewer, chromatograms from all samples should be provided. If more than 10 samples are analyzed, a minimum of 10 treated-sample chromatograms should be provided. Chromatograms of samples with unusual or inconsistent results should also be included.
(C) A minimum of three chromatograms each of control and fortified control samples for each RAC and processed fraction. These chromatograms are necessary to assess the limit of quantitation. (D) A sample calculation should be presented in which a treated sample and fortified control are taken through the entire calculation procedure. The Agency should be able to tie this calculation to a chromatogram and quantitative data included in the report. (E) All quantitative data associated with all samples should be provided so that the Agency can independently calculate the results. This would include, but not necessarily be limited to: Sample weights, extraction volumes, aliquot volumes/weights, injection volumes, final extract volumes, and peak heights/areas. Dates of each step should be specified so the treated sample data can be associated with control samples, spike recovery samples, and standards. These data can be summarized in a spreadsheet.
(F) All data associated with the calibration standards must be submitted. If a single point external standard quantitation method is used, the data for each standard should be presented so the Agency is able to calculate the residues in/on treated samples. If a calibration curve is calculated by linear regression or some other method, information for each standard should be included, along with the regression data. The Agency should have sufficient information regarding the curve calculation techniques to reproduce the standard curve. If a calibration curve is manually drawn, each calibration curve should be provided.
(G) All quantitative data associated with spike recovery samples must be submitted. This is particularly crucial if residues in/on treated samples are corrected for spike recoveries. It must be explicitly stated in the report whether results are corrected for an average recovery or the set recovery. If corrected values are reported for treated samples, apparent values should be reported as well. (6) Requirements for crop field trial studies. Crop field trials are used to assess the magnitude of the pesticide residue in/on a commodity at the time of harvest. These studies usually involve application of the pesticide to a crop in accordance with label directions in a manner which would ``* * * expose the crop to the maximum legal amount of the pesticide." The information provided is used to set pesticide tolerances. Requirements for raw data are outlined in the following list. These items should be considered in addition to the general

list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline.
(i) A summary of the weather conditions for the growing season, stating whether the weather conditions were normal, should be provided. For example, the report could state that the crop was subjected to normal temperatures with higher than average rainfall. More detailed weather data should be provided if unusual circumstances exist such as drought or hurricane which could help to explain any unusual residues found. The Agency should have sufficient information to ascertain if a field was irrigated, and the type of irrigation system used. Daily rain, wind, and temperature data need not be provided on a routine basis. (ii) Actual analysis for moisture content of potential animal feed commodities may be required by the Agency if the samples do not appear to reflect the appropriate crop stage. (7) Requirements for processing studies. Processing studies are used to assess the effect of commodity processing on the pesticide residue, i.e. if residues concentrate or are diluted. Concentration of residues may necessitate establishment of food or feed additive tolerances or maximum residue limits under section 701 of the FFDCA. Because crop field trials are an integral part of these studies, the requirements for raw data listed below should be considered in addition to those outlined in the general list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline, and the list for crop field trials.
(i) Identification of the source of the commodity which was processed (the place from which the commodity being processed was obtained).
(ii) Detailed description of processing procedures actually used and a comparison to commercial processing. (iii) Weights of sample processed and processed products. Material balances of the processes are very helpful, particularly if presented in a flow chart format.
(iv) If any phytotoxicity concerns are raised in the processing study report (precluding the use of exaggerated rates which may be necessary to produce detectable residues in/on the RAC), all data which support any statements made must be provided. (v) Moisture content of potential animal feed commodities (e.g., wet apple pomace, pineapple process residue, processed potato waste). (8) Requirements for magnitude of residue--meat, milk, poultry, and eggs studies. The Agency is concerned about pesticide residues in animal products primarily as a result of two means of exposure: Consumption of treated feeds by livestock and direct application of the pesticide to animals and their premises. Requirements for raw data are outlined in the following list. These items should be considered in addition to the general list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline.
(i) Vital statistics such as feed consumption, and milk/egg production.
(ii) A brief description of compositing procedures, such as combining milk samples from morning and afternoon milking or combining tissues from hens within a treatment group. Preparation techniques should also be specified.

(iii) If the animals are exposed to the pesticide through dermal or premise treatment, the application techniques (from applicator notes, etc.) should be provided. The Agency should have a complete understanding of the exposure.
(iv) Moisture content of feeds used in feeding studies, as well as any data on the amount of feed consumed daily by the animal. The Agency should be able to calculate the animal's exposure on dry matter and as-fed bases.
(9) Requirements for other residue chemistry studies. There are other types of residue chemistry studies which are less frequently submitted. The raw data requirements described in the general list provided under paragraphs (l)(1)(i) through (l)(1)(ix) of this guideline and in crop field trial lists typically apply to these studies as well and should be included. Listed below are some additional factors to consider for these individual studies, most of which are concerned with application of the pesticide.
(i) Magnitude of residue--water, under OPPTS 860.1400: (A) Specific description of the location and technique of the pesticide application should be provided. The description should be related to maps of the area with regard to introduction of the water (containing the pesticide residue) into water resources. (B) Sampling locations (as related to the maps) should be very specifically described. Sampling technique and timing is crucial as well.
(C) Storage stability considerations for water samples are often neglected; this information should be included. (ii) Magnitude of residue--fish, under OPPTS 860.1400: (A) Application of the pesticide to the water and introduction of the fish into the water must be thoroughly described in the field notes submitted.
(B) A complete description of the test system (static vs. dynamic, etc.) is very important in the assessment of the study. (iii) Magnitude of residue--irrigated crops, under OPPTS 860.1400:
(A) Application of the pesticide to the test system must be explicitly described.
(B) Include introduction of the pesticide into the irrigation water and frequency and method of crop irrigation. (iv) Magnitude of residue--food handling establishments, under OPPTS 860.1460:
(A) The test location and set-up must be thoroughly described. Placement of the food or commodities in the treated area should be explicitly stated. Maps of the treated areas have been very helpful to the Agency. The type of packaging and/or covers should be described. (B) The application of the pesticide must be described in detail. This would include a description of the site of application in relation to the location of the food.
(m) Raw Agricultural and Processed Commodities and Livestock Feeds Derived from Field Crops. (1) Table 1 provides a listing of all significant food and feed commodities, both raw and processed, for which residue data are collected and tolerances may be set. In addition, for feed commodities (commonly called feedstuffs) the table provides the maximum percent in the

diet for beef and dairy cattle, poultry and swine and guidance on the acceptability of label restrictions prohibiting use as a feedstuff.
(2) According to the information available, most agricultural crops and their corresponding raw agricultural and processed commodities can be, and are, fed to livestock. However, for regulatory purposes, the Agency requires residue data and livestock metabolism and feeding studies only for those feedstuffs considered to be ``significant''. In some cases, the criteria under paragraph (m)(3)(i) of this guideline were also applied to byproducts of food processing to determine if there were ``significant'' feedstuffs produced and fed to livestock. (3) The following criteria were used to decide what feedstuffs are considered ``significant'':
(i) The annual production of the crop (RAC) in the United States is more than 250,000 tons.
(ii) The maximum amount in the livestock diet is more than 10 percent.
(iii) The commodity is grown mainly as a feedstuff. (iv) The amount of a commodity (raw agricultural or processed) produced or diverted for use as a feedstuff is at least 0.04 percent of the total estimated annual tonnage of all feedstuffs available for livestock utilization in the United States. This amount is equivalent to approximately 270,000 tons on a dry matter basis. (v) Feedstuffs less than 0.04 percent of the total estimated annual tonnage of all feedstuffs available were included if one or more of the following three criteria are met: (A) The feedstuff is listed and routinely traded on the commodities exchange markets.
(B) There is regional production, seasonal considerations, or an incident history for use of the feedstuff. (C) The feedstuff is grown exclusively for livestock feeding in quantities greater than 10,000 tons on a dry matter basis (0.0015 percent of the total estimated annual tonnage of all feedstuffs available).
(4) Using these criteria for inclusion of feedstuffs in Table 1, the Agency expects to account for more than 99 percent of the available annual tonnage (on a dry-matter basis) of feedstuffs used in the domestic production of more than 95 percent of beef and dairy cattle, poultry, swine, milk, and eggs.
(5) (i) Historically, a label restriction has precluded the need for residue data, tolerances or consideration of selected commodities in the livestock diet. In reviewing the data collected on feedstuffs and public comments the Agency also reevaluated the policy of allowing as a substitute for data, a label restriction prohibiting the use (or sale) of a commodity for feeding purposes. The Agency has developed three criteria to determine whether a label restriction of a commodity from use as a feedstuff could be allowed in lieu of data. (ii) Criteria developed by the Agency are : (A) The feedstuff must remain under the control of the grower. (For example, byproducts of processing would usually not be under the control of the grower.)
(B) The crop must not be grown primarily as a feedstuff. (C) A label

restriction should cause no economic hardship. (D) The Agency's view is that there are only three cases where a label restriction should be allowed: peanut hay, soybean forage, and soybean hay.
(E) Finally, the Agency has added a statement to the table (endnote (3)) on percent dry matter requesting that the percent moisture be reported for representative samples of raw agricultural and processed commodities which are beef or dairy cattle feedstuffs. This is needed since the dietary burdens for these animals are calculated on a dry matter basis. This information will also allow the Agency to determine whether samples were harvested at the proper crop growth stage or dried to the appropriate moisture level after harvest or during processing. In the case of commodities which may have widely varying levels of moisture (e.g., apple pomace, processed potato waste), this information may be used to adjust residue levels for the establishment of tolerances. (5) A blank space in the processed commodity, feedstuff, or percent of livestock diet columns of Table 1 for a specific crop does not necessarily mean that such items are not produced from this crop, and/or used as human foods or feedstuffs, but that the Agency, at this time, does not consider these items significant in the daily dietary risk assessment of the population of the United States from pesticide use on that crop.
(6) (i) The following Table 1 lists raw agricultural and processed commodities and feedstuffs derived from field crops.

Table 1.--Raw Agricultural and Processed Commodities and Feedstuffs Derived From Crops

Note: For the electronic version of Table 1, see the Adobe PDF file. This table is not provided in ASCII format due to the size and complexity of the table. Table 1 also appears in the paper copy furnished from the Public Docket.

(ii) Table notes. The following notes are referenced in the table.
(1) Percent of Livestock Diet. For percentages of feedstuffs in livestock diets other than those listed here, contact one of the Chemistry Branches, Health Effects Division, Mail Code 7509C, Office of Pesticide Programs, Environmental Protection Agency, 401 M. St. SW., Washington, DC 20460.
(2) Percent of Livestock Diet. Maximum percent of diet on a dry weight basis for finishing beef and lactating dairy cattle, and on an as-fed basis for poultry and finishing swine (hogs). (3) % DM (percent dry matter) For beef and dairy feedstuffs, the percent moisture should be reported for representative samples of raw agricultural and processed commodities. (4) Alfalfa. Residue data are needed from a minimum of three cuttings, unless climatic conditions restrict the number of cuttings. Cut sample at late bud to early bloom stage (first cut), and/or at early (one-tenth) bloom stage (later cuts).

(5) Alfalfa seed. For registered uses on alfalfa grown for seed, residue data should be provided on seed, forage, and hay; for all other uses data should only be provided on forage and hay. (6) NU. Not used or a minor feedstuff (less than 10 percent of livestock diet).
(7) Alfalfa meal. Residue data are not needed for meal; however, the meal should be included in the livestock diet, using the hay tolerance level. Hay should be field-dried to a moisture content of 10 to 20 percent.
(8) Alfalfa silage. Residue data on silage are optional, but are desirable for assessment of dietary exposure. Cut at late bud to onetenth bloom stage for alfalfa, allow to wilt to approximately 60 percent moisture, then chop fine, pack tight, and allow to ferment for three weeks maximum in an air-tight environment until it reaches pH 4. This applies to both silage and haylage. In the absence of silage data, residues in forage will be used for silage, with correction for dry matter.
(9) Fruit. Fruit should be analyzed after removing and discarding stem, and stone or pit.
(10) Banana. Field residue data on both bagged and unbagged bananas should be provided. The required number of field trials may be split between bagged and unbagged bananas. Alternatively, one sample each of bagged and unbagged bananas may be taken from each site. Data are required on the whole commodity (including peel after removing and discarding crown tissue and stalk) for establishing tolerances. At the petitioner's discretion, residue data on just the banana pulp may be provided for purposes of dietary risk assessment. (11) Barley hay. Cut when the grain is in the milk to soft dough stage. Hay should be field-dried to a moisture content of 10 to 20 percent). Barley straw. Plant residue (dried stalks or stems with leaves) left after the grain has been harvested (threshed). (12) Barley grain, oat grain, or rice grain. Kernel (caryopsis) plus hull (lemma and palea).
(13) Bean. See Crop Group 6: Legume Vegetables under 40 CFR 180.41 for cultivars of beans. Bean seed. Dried seed for uses on dried shelled beans; succulent seed without pod for uses on succulent shelled beans (e.g. lima beans); succulent seed with pod for edible-podded beans (e.g. snap beans). Cowpea is the only bean crop considered for livestock feeding. (See cowpea). Residue data for forage and hay are required only for cowpea.
(14) Beet, sugar. Residue data may be supplied for raw sugar or refined sugar, or both raw and refined. Sugarcane. Residue data may be supplied in the same manner.
(15) Blackberry. See Crop Group 13: Berries under 40 CFR 180.41 for cultivars of blackberries.
(16) Buckwheat grain. Seed (achene) plus hull. (17) Cabbage fresh, with wrapper leaves. Entire cabbage head with obviously decomposed or withered leaves removed. In addition, residue data on cabbage head, without wrapper leaves, are desirable particularly when a more accurate assessment of dietary exposure is necessary.

(18) Carrot culls. Data for raw agricultural commodities will cover residues on culls.
(19) Clover forage. Cut sample at the 4 to 8 inch to prebloom stage, at approximately 30 percent DM. Clover hay. Cut at early to full bloom stage. Hay should be field-dried to a moisture content of 10 to 20 percent. Residue data for clover seeds are not needed. (20) Clover silage. Residue data on silage are optional, but are desirable for assessment of dietary exposure. Cut sample at early to one-fourth bloom for clover, allow to wilt to approximately 60 percent moisture, then chop fine, pack tight, and allow to ferment for 3 weeks maximum in an air-tight environment until it reaches pH 4. This applies to both silage and haylage. In the absence of silage data, residues in forage will be used for silage, with correction for dry matter. (21) Coffee. Residue data are required on the green bean, the roasted bean, and on instant coffee. Tolerances on the green bean will be established under section 408 of the FFDCA. Maximum residue limits on the roasted bean and instant coffee will be established under section 701 of the FFDCA if residues exceed those on the green bean. The green bean is the dried seed of the coffee bean. (22) Field corn forage. Cut sample (whole aerial portion of the plant) at late dough/early dent stage (black ring/layer stage for corn only). Sorghum forage. Cut sample (whole aerial portion of the plant) at soft dough to hard dough stage). Forage samples should be analyzed as is, or may be analyzed after ensiling for 3 weeks maximum, and reaching pH 5 or less, with correction for dry matter. (23) Corn stover. Mature dried stalks from which the grain or whole ear (cob + grain) have been removed; containing 80 to 85 percent DM. Sorghum stover. Mature dried stalks from which the grain has been removed; containing approximately 85 percent DM. (24) Aspirated grain fractions (previously called grain dust). Dust collected at grain elevators for environmental and safety reasons. Residue data should be provided for any postharvest use on corn, sorghum, soybeans, or wheat). For a preharvest use after the reproduction stage begins and seed heads are formed, data are needed unless residues in the grain are less than the limit of quantitation of the analytical method. For a preharvest use during the vegetative stage (before the reproduction stage begins), data will not normally be needed unless the plant metabolism or processing study shows a concentration of residues of regulatory concern in an outer seed coat (e.g. wheat bran, soybean hulls).
(25) Corn starch. Residue data for starch will be used for corn syrup. Petitioners may also provide data on syrup for a more accurate assessment of dietary exposure.
(26) Corn milled byproducts. Use residue data for corn drymilled processed commodities having the highest residues, excluding oils.
(27) Sweet corn. Residue data on early sampled field corn should suffice to provide residue data on sweet corn, provided the residue data are generated at the milk stage on kernel plus cob with husk removed and there are adequate numbers of trials and geographical representation from the sweet

corn growing regions.
(28) Sweet corn (K + CWHR). Kernels plus cob with husks removed. (29) Sweet corn forage. Samples should be taken when sweet corn is normally harvested for fresh market, and may or may not include the ears. Petitioners may analyze the freshly cut samples, or may analyze the ensiled samples after ensiling for 3 weeks maximum, and reaching pH 5 or less, with correction for percent dry matter. (30) Sweet corn cannery waste. Includes husks, leaves, cobs, and kernels). Residue data for forage will be used for sweet corn cannery waste.
(31) Cotton gin byproducts (commonly called gin trash). Include the plant residues from ginning cotton, and consist of burrs, leaves, stems, lint, immature seeds, and sand and/or dirt. Cotton must be harvested by commercial equipment (stripper and mechanical picker) to provide an adequate representation of plant residue for the ginning process. At least three field trials for each type of harvesting (stripper and picker) are needed, for a total of six field trials. (32) Cowpea forage. Cut sample at 6 inch to prebloom stage, at approximately 30 percent DM. Cowpea hay. Cut when pods are one-half to fully mature. Hay should be field-dried to a moisture content of 10 to 20 percent.
(33) Crownvetch forage. Cut sample at 6 inch to prebloom stage, at approximately 30 percent DM. Crown vetch hay. Cut at full bloom stage. Hay should be field-dried to a moisture content of 10 to 20 percent.
(34) Grass. Zero day crop field residue data for grasses cut for forage should be provided unless it is not feasible, e.g. preplant/ preemergent pesticide uses. A reasonable interval before cutting for hay is allowed. Grass forage. Cut sample at 6 to 8 inch to boot stage, at approximately 25 percent DM. Grass hay. Cut in boot to early head stage. Hay should be field-dried to a moisture content of 10 to 20 percent. Grasses include barnyardgrass, bentgrass, Bermudagrass, Kentucky bluegrass, big bluestem, smooth bromegrass, buffalograss, reed canarygrass, crabgrass, cupgrass, dallisgrass, sand dropseed, meadow foxtail, eastern gramagrass, side-oats grama, guineagrass, Indiangrass, Johnsongrass, lovegrass, napiergrass, oatgrass, orchardgrass, pangolagrass, redtop, Italian ryegrass, sprangletop, squirreltailgrass, stargrass, switchgrass, timothy, crested wheatgrass, and wildryegrass. Also included are sudangrass and sorghum forages and their hybrids. For grass grown for seed only, PGIs (pregrazing intervals) and PHIs (preharvest intervals) are acceptable. Residue data may be based on the regrowth after harvesting the seed.
(35) Grass silage. Residue data on silage are optional, but are desirable for assessment of dietary exposure. Cut sample at boot to early head stage, allow to wilt to 55 to 65 percent moisture, then chop fine, pack tight, and allow to ferment for 3 weeks maximum in an airtight environment until it reaches pH 4. In the absence of silage data, residues in forage will be used for silage, with correction for dry matter.
(36) Herbs. Consist primarily of leaves, stems, and flowers and are

marketed fresh (succulent) or dried). See Crop Subgroup 19-A under 40 CFR 180.41 for listing of herbs.
(37) Hops, cones, dried. According to PR Notice 93-12 (December 23, 1993), dried hops will be considered as a raw agricultural commodity for regulatory purposes. Residue data are needed for dried hops only. (38) Lespedeza forage. Cut sample at 4 to 6 inch to prebloom stage, at 20 to 25 percent DM. Lespedeza hay: Annual/Korean. Cut at early blossom to full bloom stage. Sericea. Cut when 12 to 15 inches tall. Hay should be field-dried to a moisture content of 10 to 20 percent.
(39) Lettuce, fresh, with wrapper leaves. Entire lettuce head with obviously decomposed or withered leaves removed. In addition, residue data on lettuce head, without wrapper leaves, are desirable particularly when more accurate assessment of dietary exposure is necessary.
(40) Lettuce, leaf. Residue data should be on samples with obviously decomposed or withered leaves removed. (41) Millet forage. Cut sample at 10 inches to early boot stage, at approximately 30 percent DM. Millet hay. Cut at early boot stage or approximately 40 inches tall, whichever is reached first. Hay should be field-dried to a moisture content of 10 to 20 percent. Millet includes pearl millet.
(42) Millet grain. Kernel plus hull (lemma and palea). Pearl millet grain. Kernel with hull (lemma and palea) removed. (43) Millet straw. Data are required for proso millet only. Proso millet straw. Plant residue (dried stalks or stems with leaves) left after the grain has been harvested. (44) Millet flour. Not produced significantly in the United States for human consumption. Residue data are not needed at this time. (45) Mung bean. Data on mung bean covers sprouts except when the pesticide is used on the sprouts per se. (46) Muskmelon. Includes cantaloupe, casaba, crenshaw, etc. See Crop Group 9: Cucurbit Vegetables under 40 CFR 180.41 for other cultivars of muskmelons.
(47) Nuts. Includes Crop Group 14: Tree Nuts nder 40 CFR 180.41, except for almonds. Pistachio is under consideration to be added to Crop Group 14. Residue data for tree nuts may be used to support uses on pistachio. See Crop Group 14 for a listing of nuts. Also see almonds. Almond hulls are considered a significant feedstuff. Hulls from other tree nuts are not considered significant feedstuffs. (48) Oats forage. Cut sample between tillering to stem elongation (jointing) stage. Oats hay. Cut sample from early flower to soft dough stage. Hay should be field-dried to a moisture content of 10 to 20 percent. Oats straw. Cut plant residue (dried stalks or stems with leaves) left after the grain has been harvested (threshed). (49) Parsley. Fresh parsley is included in Crop Group 4: Leafy Vegetables under 40 CFR 180.41. Dried parsley is included in Crop Subgroup 19A: Herbs under 40 CFR 180.41. (50) Pea. Residue data for forage and hay are required for cowpea. (See cowpea). Residue data for vines and hay are required for field peas only. (See pea, field).
(51) Pea, succulent. Succulent seed with pod for edible-podded peas (e.g.

snow peas); succulent seed without pod for uses on succulent shelled peas (e.g. English peas).
(52) Pea seed. Mature dried seed for uses on dried shelled peas. (53) Pea, field. Does not include the canning field pea cultivars used for human food) Includes cultivars grown for livestock feeding only such as Austrian winter pea. Field pea vines. Cut sample anytime after pods begin to form, at approximately 25 percent DM) Field pea hay. Succulent plant cut from full bloom through pod formation). Hay should be field-dried to a moisture content of 10 to 20 percent. (54) Pea, field, silage. Use field pea vine residue data for field pea silage with correction for dry matter. (55) Peanut hay. Peanut hay consists of the dried vines and leaves left after the mechanical harvesting of peanuts from vines that have been sun-dried to a moisture content of 10 to 20 percent. (56) (R): Label restrictions against feeding may be allowed; e.g. Do not feed green immature growing plants to livestock, or Do not harvest for livestock feed.
(57) Pepper. Nonbell pepper includes chili pepper. (58) Pimento. The official name adopted by the Georgia Pimento Growers Association.
(59) Pineapple process residue (also known as wet bran). A wet waste byproduct from the fresh-cut product line that includes pineapple tops (minus crown), bottoms, peels, any trimmings with peel cut up, and the pulp (left after squeezing for juice); it can include culls. (60) Plantain. Banana tolerance will cover plantain. (61) Potato granules/flakes. Residue data may be provided for either.
(62) Processed potato waste. Tolerance levels for wet peel should be used for dietary burden calculations. Residue data may be provided from actual processed potato waste generated using a pilot or commercial scale process that gives the highest percentage of wet peel in the waste.
(63) Rapeseed meal. Residue data are not needed for rapeseed oil since it is produced for industrial uses and is not an edible oil. The edible oil is only produced from canola. (See canola.) (64) Rape greens. A commodity listed in Crop Group 5: Brassica (Cole) Leafy Vegetable Group under 40 CFR 180.41. (65) Rice straw. Stubble (basal portion of the stems) left standing after harvesting the grain.
(66) Rye forage. Cut sample at 6 to 8 inch stage to stem elongation (jointing) stage, at approximately 30 percent DM. Rye straw. Cut plant residue (dried stalks or stems with leaves) left after the grain has been harvested (threshed).
(67) Rye grain or wheat grain. Kernel (caryopsis) with hull (lemma and palea) removed.
(68) Sorghum flour. Residue data are not needed at this time since sorghum flour is used exclusively in the United States as a component for drywall, and not as either a human food or a feedstuff. However, because 50 percent of the worldwide sorghum production goes toward human consumption, data may be needed at a later date. (69) Sorghum, sweet. Sweet sorghum commodities (i.e., seed and forage) will be covered by the sorghum grain tolerances.

(70) Soybean forage. Cut samples at 6 to 8 inches tall (sixth node) to beginning pod formation, at approximately 35 percent DM. Soybean hay. Cut samples at mid-to-full bloom stage and before bottom leaves begin to fall or when pods are approximately 50 percent developed. Hay should be field-dried to a moisture content of 10 to 20 percent.
(71) Soybean silage. Residue data on silage are optional. Harvest sample when pods are one-half to fully mature (full pod stage). In the absence of silage data, residues in forage will be used for silage, with correction for dry matter . (72) Spices. Include aromatic seeds, buds, bark, berries, pods, and roots consumed and marketed primarily in their dried form. See Crop Subgroup 19-B under 40 CFR 180.41 for listing of spices. (73) Sugarcane bagasse. Information indicates that sugarcane bagasse is mainly used for fuel. Residue data will not be needed at this time, but may be needed at a later date. (74) Sugarcane molasses. Residue data are needed for blackstrap molasses.
(75) Tea. Residue data are required on plucked (or freshly picked leaves, dried tea, and instant tea. Tolerances on plucked tea leaves will be established under section 408 of FFDCA. Maximum residue limits on dried tea and instant tea will be established under section 701 of the FFDCA if residues exceed those on the plucked tea. (76) Tomato paste. Residue data on tomato paste cover tomato processed products (e.g. sauce, juice, catsup), except tomato puree which covers canned tomatoes.
(77) Trefoil forage. Cut sample at 5 to 10 inch or early bloom stage, at approximately 30 percent DM. Trefoil hay. Cut at first flower to full bloom. Hay should be field-dried to a moisture content of 10 to 20 percent.
(78) Vetch forage. Cut sample at 6 inch to prebloom stage, at approximately 30 percent DM. Vetch hay. Cut at early bloom stage to when seeds in the lower half of the plant are approximately 50 percent developed. Hay should be field-dried to a moisture content of 10 to 20 percent. Vetch does not include crownvetch. (79) Wheat forage. Cut sample at 6 to 8 inch stage to stem elongation (jointing) stage, at approximately 25 percent DM. Wheat hay. Cut samples at early flower (boot) to soft dough stage. Hay should be field-dried to a moisture content of 10 to 20 percent. Wheat straw. Cut plant residue (dried stalks or stems with leaves) left after the grain has been harvested (threshed).
(80) Wheat. Includes emmer wheat and triticale. No processing study is needed for a specific tolerance on emmer wheat. (81) Wheat milled byproducts. Use highest value for wheat middlings, bran, and shorts.
(n) References. The following references should be consulted for additional background material on this test guideline. (1) Environmental Protection Agency. Pesticide Reregistration Rejection Rate Analysis--Residue Chemistry; Follow-up Guidance for: Generating Storage Stability Data; Submission of Raw Data; Maximum Theoretical Concentration Factors; Flowchart Diagrams. EPA Report No. 737-R-93-001, February 1993.
(2) Environmental Protection Agency. Pesticide Reregistration Rejection

Rate Analysis--Residue Chemistry; Follow-up Guidance for: Updated Livestock Feeds Tables; Aspirated Grain Fractions (Grain Dust); A Tolerance Perspective; Calculating Livestock Dietary Exposure; Number and Location of Domestic Crop Field Trials. EPA Report No. 737-K-94-001, June 1994.
(3) Environmental Protection Agency. Pesticide Reregistration Rejection Rate Analysis--Residue Chemistry. EPA Report No. 738-R-92-001, June 1992.
(4) Environmental Protection Agency. FIFRA Accelerated Reregistration--Phase 3 Technical Guidance. EPA Report No. 540/09-90- 078, December 1989.
(5) Environmental Protection Agency. Pesticide Regulation (PR) Notice 96-1, Tolerance Enforcement Methods--Independent Laboratory Confirmation by Petitioner, February 7, 1996.