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OECD GUIDELINE FOR THE TESTING OF CHEMICALS

Magnitude of the Pesticide Residues in Processed Commodities

INTRODUCTION

1. A wide range of raw agricultural commodities (RAC) are processed before they are consumed. Processing studies are normally used to determine residue levels in primary processed commodities following pesticide application under label conditions likely to lead to maximum residues. Such situations include pre- or post-harvest pesticide use and direct animal treatment or veterinary use. This guideline does not include simple peeling or washing practices, nor does it include fodder production, as these practices are generally addressed in the supervised residue field trials.

PURPOSE

2. Studies on the magnitude of residues in processed commodities provide data on the transfer of residues to different processed commodities from the raw agricultural commodity (RAC). Studies on the magnitude of residues are conducted in order to quantify levels of residues in processed commodities and to provide the distribution of residues (active ingredient, and/or metabolites, degradation products) in various processed products resulting from the processing of a commodity. This information about dilution and concentration of residues and the estimation of processing factors (the ratio of residue levels in processed commodities to those in the raw agricultural commodity) is used to:

- conduct refined dietary exposure assessments with primary processed products to assess consumer safety;
- provide results on residues in commodities that may be used as animal feedstuffs and thus to allow a more realistic calculation of the dietary burden of livestock;
- establish MRLs for processed commodities; and
- monitor compliance with the RAC MRL.

The procedures that are used to produce processed commodities are diverse and varied. This guideline describes how to plan and carry out processing studies.

APPLICABILITY OF PROCESSING STUDIES

3. This guideline applies to RACs of plant origin. It also applies to RACs of animal origin in cases of direct animal treatment or veterinary use. Applicability of studies on the magnitude of residues in processed commodities depends upon the importance of a processed product in the human and/or animal diet; the possibility of residue levels in processed foods/feeds exceeding the level in a RAC; the level of residue in the plant or plant product to be processed (RAC); the physical - chemical properties of the active ingredient or relevant metabolites; and the possibility that degradation products of toxicological significance may be found in animal or plant products after processing.

GENERAL CONSIDERATIONS

4. Definitions:

a) The term “Primary Processed Commodities” means the products - resulting from the application of physical, chemical or biological processes or combinations of these to a “primary food commodity” - intended for direct sale to the consumer, for direct use as an ingredient in the manufacture of food or for further processing. A primary processed commodity is derived from mechanical or chemical processing of the RAC and is not a multi-component product (Codex).

b) Critical GAP (*c*GAP) is the treatment at the maximum of good agricultural practices that may lead to the highest residues of the pesticide for a specific crop/pesticide combination.

5. Pesticide residues to be measured in processing studies are determined by the residue definition which is derived from studies on the nature of the residue in processing and/or in plant and livestock. Processing factors are then determined for use in further assessments.

6. Processing studies should measure metabolites and degradation products which are included in the residue definition as well as degradation products identified in the "Nature of the Residue in Processed Commodities - High Temperature Hydrolysis" study which are deemed significant, based on the level found in the study and their toxicological significance.

7. Processing studies should simulate industrial or domestic practices as closely as possible. RACs used in processing studies should contain field-treated (incurred) quantifiable residues, at sufficient levels that concentration/dilution factors for the various consumed products and non-consumed intermediates (for example, cooking water) can be determined. This may require field treatment at exaggerated application rates or other appropriate measures such as shortened PHI (if there is no indication that the composition/behaviour will change) to obtain sufficient residue levels for processing studies. Processing studies utilising spiked samples are not acceptable.

8. The processing factor (P*f*) that originates only from residues of the same single compound in the RAC is calculated as follows:

$$Pf = \frac{\text{residue level in processed commodity}}{\text{residue level in the RAC or commodity to be processed}}$$

9. For each field test site sampled, the residue level in the processed commodity is compared to that in the RAC. From the results of processing of RACs obtained from two independent field test sites for a processing study, the mean value of the two P*f* is calculated to give the processing factor. This factor is valid for the combination procedure/commodity which was investigated in the processing study. In case of three or more processing tests, the processing factor is the median of the single factors from each test.

10. If the residue definition for enforcement purposes in processed products differs from that in the RAC, the processing factor should be calculated taking into account the molecular weights of the different substances. For the calculation, three different cases have to be considered and are described in the OECD Guidance for Residues in Processed Commodities. (Insert link when available)

11. The FAO Manual states that "if the processing factors from two trials are irreconcilable, e.g. 10-fold different, the mean is inappropriate because it would not represent either process. In this case it is preferable to choose one of the values as being representative. The highest processing factor should be

chosen as the default (conservative) value if there is no reason to choose one or the other." Also, in such cases, the trials within the study should be reviewed with care to determine whether the results are valid or whether two completely different procedures were compared.

12. Where there is a large difference in the results of two trials within a processing study it may be necessary to conduct an additional trial for that procedure. It is well known that results of two processing trials vary within a certain range. A difference of 50% is a practical estimate of maximum variation for two trials. When the resulting processing factors from the two trials for the same processing procedure differ with respect to the main processed products by more than 50%, a third trial may be necessary to derive a consistent processing factor. The difference of 50% is calculated as follows:

$$\frac{Pf \text{ (high value)} - Pf \text{ (low value)}}{Pf \text{ (high value)}} \geq 0.5$$

13. Before setting up a third trial for a processing procedure, the existing trials should be investigated to determine which factors influence the level of residues in the processed commodity and thus to choose the realistic worst case conditions for the third trial.

14. Important conclusions concerning the behaviour of the active ingredient and/or metabolites during processing can be drawn from the distribution coefficients for *n*-octanol/water, hydrolysis stability, heat stability and solubility behaviour. For example, when the log P_{ow} is greater than three, it can be assumed that the residue will likely be concentrated in oil or solids like meal, whereas high water solubility indicates that residues may be expected in juices. For example: the extremely high potential concentration factors for citrus oil (P*f* = 1000) and mint oil (P*f* = 330) should be considered.

15. In processes where dehydration is the path from RAC to processed commodity, a simple calculation based on the loss of water is sufficient for derivation of default generic processing factors to assess potential for exceedance of a RAC MRL. Such factors represent the maximum theoretical transfer of residues to the dried product and in reality, the transfer is often less. While these factors may be used to perform a preliminary dietary exposure assessment, it is not considered good practice to establish MRLs for processed commodities based on default dehydration factors (% dry matter or %DM). Processing studies are used for enforcement purposes and refined dietary exposure assessment in lieu of the default factors.

16. Default factors are not applicable even for preliminary dietary exposure assessment in cases where the process generates a relevant compound. An estimate of the yield from parent compound (in lieu of a separate processing factor) is needed for the metabolite/degradation product in cases where the procedure generates a relevant compound (for example, dithiocarbamates produce ETU upon dehydration of the RAC).

Situations in which a study may or may not be necessary

17. Table 1 describes two categories of processing procedures. Category 1 procedures involve well-defined procedures typically practiced on a large industrial scale for major commodities. Most regulatory authorities consider studies for these procedures essential. A corresponding domestic use may also exist and be covered by the industrial use. Category 2 procedures are a mixture of domestic (or home) and industrial procedures. These types of processing studies, while encouraged, are often considered optional by some regulatory authorities. The studies are particularly useful in refining dietary exposure assessments.

18. When no residues are found at or above the appropriate LOQ in the RAC at the *c*GAP from **all** supervised field trials, no processing studies are necessary for the procedures identified in Category 2 of Table 1. Likewise, no processing studies are necessary for the procedures identified in Category 1 of Table 1 under the above conditions provided that, in addition, no significant potential for concentration in processed food is likely to occur. Potential to concentrate is based on three considerations:

a) The properties of the pesticide – These must predict that the pesticide (and metabolites, as appropriate) will not concentrate in the processed commodity. For example, a water soluble pesticide (e.g. water solubility >0.5 mg/L) would not be expected to concentrate in the processed oil from an oilseed but the same pesticide might concentrate in the juice from an orange.

b) The theoretical concentration factor – This is based on the relative percentages (by mass) of processed fractions obtained from a particular commodity.

c) Extremely high concentration factor – The consideration of processing studies for commodities with no quantifiable residues when the crop is treated according to *c*GAP is particularly important for commodities with extremely high theoretical concentration factors. These include mint to mint oil, citrus to citrus oil, and corn to corn oil. In those cases where residues in citrus peel are below the LOQ following application at a rate 5 times the proposed rate, data on citrus oil are not required.

19. If the properties of the pesticide (and/or metabolites as appropriate) indicate that it might concentrate in a given processed fraction, then a processing study may be necessary. The pesticide should be applied to the crop at an exaggerated rate, up to five times, to attempt to generate a commodity with quantifiable residues, but not if phytotoxicity occurs. This commodity, if it contains quantifiable residues, would be processed. If it does not contain quantifiable residues, a processing study would not be needed.

20. The need for processing studies for commodities in procedures from Category 1 of Table 1 where no quantifiable residues are found at *c*GAP conditions may vary with national or regional governments, and the applicant should consult the appropriate regulatory authority.

Processing procedure types and extrapolation

21. Commodities fall into natural types with respect to processing. These commodity types may or may not align with field trial crop groupings globally. Additional justification for types of extrapolation follows.

22. For commodity belonging to the same commodity type and undergoing the same processing procedure it is assumed that the results from studies from one commodity can be extrapolated to the other commodities of this type, including all similar processed commodities within the procedure. For example, results from processing oranges to orange juice and pomace can be extrapolated to the processing of other citrus fruits.

23. Oilseeds may be considered to fall into two types: low (approximately 20%) and high (approximately 50%) oil content. Examples of oil content for different oilseeds can be found in the Guidance for Residues in Processed Commodities. Translation of the processing factor from an oil seed with 50% oil to an oilseed with 10% oil could theoretically underestimate the concentration by a factor of five for the seed with low oil content when processing RACs from crops (or RACs for post harvest uses) treated with fat-soluble compounds. However, an extrapolation from a low oil content oilseed to one with a high oil content would be acceptable, although it might be an overestimate.

24. Furthermore, in some cases it is proposed to extrapolate the results of a processing study conducted with one crop to other crops belonging to another crop group where the same type of processing procedure is used. As in the previous example, the results of a processing study to prepare orange juice from oranges might be translated to other tropical fruit juices. In these cases, an extrapolation to other processed commodities occurring during the same process may or may not be extrapolated. The possibility of extrapolating should be carefully examined and discussed with appropriate regulatory authorities. The possible extrapolations are given in Table 1.

25. The crops mentioned in column 4 of Table 1 are only examples of some important crops for the respective processing procedure**.** The selection of the crop/RAC depends on the use pattern of the pesticide, the range of crops for which registration is being sought in a number of countries, and its physical-chemical properties which affect its behaviour as described above.

Table 1: Processing Procedure Types and Recommended Extrapolations Using Typical RACs

Type[1)]	**Processing procedure**	**Explanations**	**Examples of typical crop/RAC**	**Extrapolations**	**Domestic or Industrial**[2)]
Category 1(Major Industrial Procedures)					
II	Preparation of fruit juice	Also covering pomace or dried pulp (byproducts) as an animal feed	orange apples grapes (see #V also)	orange → citrus (juice, feed), tropical fruits (juice only) apple→pome fruit, stone fruit (juice, feed) grapes→small berries (juice, feed)	D/I
V	Preparation of alcoholic beverages	Fermentation Malting Brewing Distillation	Grapes (wine) Rice Barley Hops Other Cereals (wheat, maize, rye) Sugar cane	Grapes[3)]→ all wine-producing RACs except rice Rice (beer, wine) →None Barley[4)]→all beer-producing RACs (except rice and hops) Barley→all whiskey-type producing RACs	D/I
VII	Preparation of vegetable juice	Includes preparation of concentrated juices, e.g., tomato puree and paste	Tomatoes Carrot	Tomato→all vegetables	D/I
X	Preparation of oil	Pressing or extraction including meal or press cake used as animal feed	Rapeseed (canola) Olives Maize (Corn)	1)Solvent extraction (crushing): Olive→None cottonseed↔soybean→ rapeseed (canola)↔ other oilseeds 2)Cold press: Olive→None cottonseed↔soybean→ rapeseed (canola)↔ other oilseeds	I

Type[1)]	Processing procedure	Explanations	Examples of typical crop/RAC	Extrapolations	Domestic or Industrial[2)]
				3)Milling (wet&dry): Maize→None	
XI	Distribution on milling	Including bran and gluten used as animal feed. Other grain fractions used as feeds.	Wheat Rice Maize (corn)	Wheat→all small grains except rice (oats, barley, triticale, rye) Rice→wild rice Maize (corn, dry milling)→ sorghum	I
XIV	Silage production	Important animal feed items.	Beets Pasture Grass / alfalfa	Beets (pulp) → roots and tubers Pasture grass / alfalfa silage → all green plant silage	I
XII	Preparation of sugar	Molasses and bagasse (used as animal feed) are only items that might contain concentrated residues. Other processed commodities, such as sugar, should also be evaluated.	sugar beets, sugar cane, sweet sorghum	Cane↔beet (only refined sugar)	I
Category 2 (Other Industrial Procedures and Domestic or Home Procedures					
XIII	Infusions and extractions	Infusions, including green and black tea. Roasting and extraction (including instant coffee)	Tea Cacao Coffee	None	D/I
III	Preparation of canned fruit		Canned: Apple/Pear Cherries/Peach Pineapple	Any fruit canned with skin →all canned fruits	D/I
IV	Preparation of other fruit products (primary procedures only)	Includes production of marmalade, jam, jelly, sauce/puree[5)]	Pome fruit Stone fruit Grape Citrus (orange)	Any one ~~fruit~~other major fruits	D/I
VI	Cooking vegetables, pulses and grains in water (including steaming)		Carrots Beans/peas (dry) Beans/peas (succulent) Potatoes Spinach Rice [polished (white) or husked (brown)]	Spinach → leafy vegetables, brassica vegetables (<20 minutes) Potatoes → root, tuber, bulb vegetables, fresh legumes (>20 minutes) Rice →all grains	D
VIII	Preparation of canned vegetables		Common (green or snap) bean Corn (sweet) Pea (garden, succulent) Potato Spinach Beet (garden, table)	Common bean, corn, pea, or spinach vegetables Potato → sweet potato	D/I

Type[1)]	Processing procedure	Explanations	Examples of typical crop/RAC	Extrapolations	Domestic or Industrial[2)]
			Tomato Pulses (pea or bean)		
IX, XVIII	Miscellaneous preparations of other vegetable products	Frying Microwaving Baking	Potatoes	Potatoes→all vegetables (microwaving) Potatoes→all vegetables (frying and baking)	D/I
XV	Processing of products of animal origin including preparation of meat and fish[6)]	Churning Boiling/poaching Baking/smoking Frying Fermentation	Milk Eggs Meat Fish	None	D/I
XVI	Dehydration	Removal of water	Fruits (esp grapes, plums) Vegetables Potatoes Grasses	None	I
XVII	Fermentation of soybeans, rice and others (except alcoholic beverages)	Fermentation	Cabbage Soya (soybean) Rice	None	D/I
XIX	Pickling	Brining or corning, the procedure of preserving food by anaerobic fermentation in salt solution	Cucumber Cabbage	Cucumbers→all vegetables	D/I

1) Complete list of Types may be found in the Annex 1 of "Guidance for Residues in Processed Commodities.
2) For further explanation see paragraph 31.
3) Processing studies are necessary for both red and white wine grapes.
4) Although beer is not considered a primary processed commodity, being a multi-component product with multiple steps, it is an important processed commodity and the procedures to prepare it should be included in Category 1.
5) The procedures for marmalade, jam and jelly are not considered primary so a processing study may not be conducted; since the amount of sugar used in these procedures is significant (30-60% sugar), any calculations to determine a processing factor in place of an actual study, should be based on 50% fruit content, or a processing factor of 0.5 for that step in the procedure (sugar addition step: fruit RAC residue X 0.5 = marmalade residue).
6) Animal RAC processing conducted only if a veterinary use (direct animal treatment) is requested.

26. The field phase of the processing study should follow the appropriate, existing regional guidelines for conducting field trials. A harmonised OECD guideline for conducting field trials is being developed and, when final, will be the basis for the field portion of the processing studies. The analytical phase of the processing study will comply with the OECD "Guidance Document on Pesticide Residue Analytical Methods".

CONDUCT OF STUDIES

Test conditions

27. Processing studies representative of the potential uses of a given pesticide on crops in domestic and industrial preparations of food/feed are usually needed. At least two independent trials, with RAC samples from two separate field sites, are necessary for each processing procedure (domestic/industrial) to be conducted. The same GLP facility may be used for processing both trials.

28. Two trials are not sufficient in those situations where two or more significantly different commercial procedures are practiced for a given commodity. For example, the two independent trials are not sufficient in the cases of wine making, the milling of corn, and oil production. The preparation of white wine is different from the preparation of red wine since red wine processing may involve heating and inclusion of skins. Therefore, processing studies with at least two tests on white wine preparation and two tests on red wine preparation are necessary. The milling of corn involves two completely different procedures, wet or dry milling. Also in this case, at least two tests with wet milling and two tests with dry milling are required. For oil production, if both solvent extraction and cold press procedures are used for the crop of interest, then at least two tests of each are required.

Test substance

29. RAC samples used in processing studies should contain quantifiable residues – ($\geq$ LOQ), but preferably up to at least 0.1 mg/kg or 10 times the LOQ – so that processing factors for the various processed products can be determined. Only RACs containing incurred residues shall be used for processing.

30. The residues in the sample immediately prior to processing must be determined and reported. At least two replicate samples of the RAC should be analysed. The actual weights for the RAC samples to be processed should be reported.

Processing technology

31. The technology to be used in processing studies should correspond as closely as possible to the actual conditions that are normally used in practice. A distinction should therefore be made between domestic and industrial processing procedures. Thus, processed products that are prepared domestically (e.g. cooked vegetables) should be produced using the equipment and preparation techniques that are normally used in the home. On the other hand, industrially produced processed products (e.g. cereal fractions, preserves, fruit juices, sugar, oils) should be produced using commercially representative technology, including cleaning steps, even when there is a corresponding domestic procedure. A flow chart and/or SOP describing the main process are highly recommended for both domestic and industrial processing.

Products to be covered

32. In principle, for every crop having residues and being processed, a set of processing studies should be conducted. It should be possible to extrapolate the processing factor for the given pesticide to all crops within the given group undergoing the same procedure. The possibility of extrapolating this factor to all crops undergoing the same procedure should be carefully examined and discussed with appropriate regulatory authorities (see Table 1). The aim of Annex 1 of the Guidance for Residues in Processed Commodities is to provide users with a compilation of processed commodities important for the calculation of dietary exposure to humans and animals. For this reason, commodities from the OECD feed table as well as major

processed commodities used as foods are incorporated. Nevertheless, it is not intended to give a complete list of all processed commodities from all crops under all circumstances.

Sampling

33. Detailed information on the types of processed samples to be taken for analysis is given in Annex 1 of Guidance for Residues on Processed Commodities. RAC samples for analysis must be taken from the bulk sample immediately prior to processing and stored frozen before subsequent analysis. Samples should be taken at the end of the processing procedure and stored under frozen conditions in inert sealed containers, if they need to be stored. Where intermediate samples are required for processing factors, these should be taken at appropriate points within the process and stored frozen as well. Some non-homogeneous samples, such as must in wine processing, lend themselves to having replication of sampling to ensure a representative residue value. Replicate sampling and analyses are always encouraged. The total weight of each of the individual processed fractions should be reported.

Sample analysis

34. The analytical method including sample extraction and clean-up procedures should be described in detail or referenced and should comply with the OECD Guidance Document on Residue Analytical Methods. Spiked samples should be run concurrently with those from the processing study to validate the method. The validation of the analytical method should target an LOQ that is appropriate considering the toxicity of the components of the residue definition and the need of the data for use in dietary exposure assessment.

Storage stability data

35. For pre-harvest uses, samples should be processed as soon as possible following harvest in order to keep the integrity of the RAC. For post-harvest uses, (e.g. on cereal grains), processing should take place after an interval simulating commercial storage times, e.g. 3-6 months after treatment, to allow the residues to "age", which may influence the profile of the residues in processed commodities. As outlined in the OECD Test Guideline "Stability of Pesticide Residues in Stored Commodities, if there is no observed decline of residues across the range of the five different crop categories (including animal matrices, if applicable) from the RAC storage stability study, then specific residues freezer stability data for processed foods will not be needed. However, if instability is shown after a certain length of storage, the applicant should ensure that any commodities (RAC, animal tissue or processed commodity) are analysed within the demonstrated time period for stable storage. For samples not analysed within the approved storage interval for the respective RAC, storage stability data should be generated to provide sufficient evidence that there was no significant degradation of the components of the residue definition between sampling and analysis.

CONSIDERATIONS FOR DATA REPORTING

36. The following elements should be considered during the design, conduct and reporting of the study.

Summary / Introduction

- Core processing procedures employed and the rationale for the selection of these procedures.
- The overall experimental procedure employed should include a discussion, if applicable, of unusual experimental problems encountered, attempts made to alleviate these problems which

resulted in deviations from the intended test protocol and the effects, if any, of those deviations on the results of the study.

- Summary of key results: residues in different processed products, processing factors, preferential accumulation in certain processed products, highest residues.
- Evaluation of these results.
- Any anomalies of the study, an evaluation of their relevance with reference to the objective.

Objective

A description of the aims of the study in detail including the questions to be dealt with in the study.

Test material

The pesticide plant protection product (or formulation) should be identified by:

- Type of formulation,
- Content of active ingredient(s),
- Source and purity.

The pesticide active ingredient and/or metabolites which are used in the processing study should be identified by:

- Chemical name (IUPAC),
- Common name (ANSI, BSI, IS0) (if available),
- Chemical Abstracts Service (CAS) name and number.
- The source and purity of each compound should be specified when spiked samples are prepared for analytical method validation and/or storage stability studies, if necessary.
- Chemical structure(s) for the active ingredient and metabolites constituting the residue should be provided and a cross reference of all different developmental or experimental names should be provided in either an overview document or as an appendix to the study. Certificates of analysis describing the purity and the identity of standards used in the identification process should be provided if available.

Test site/process

- Include a description of the test facility, indicating their location.
- Rationale for the kind of procedure – domestic or industrial.

RAC to be processed

- RAC and pesticide use history preceding the study, either by citing the entire residue study or by describing the residue study itself.
- Codex Commodity names, or the nearest Codex Commodity names equivalent to the commodity descriptions being used.

- Sampling: sample weights.
- Preparation of RAC prior to processing including storage conditions (including shipping condition, if applicable) and duration.

Processing

- Detailed description of the procedure including a flow chart where possible.
- Sampling: weights of processed fractions.
- Description of sampling points and state of the commodity sampled.

Analytical method

- Describe methods fully or reference them if previously submitted, including any method validation, recovery and LOQ data. Preparation and handling of the sample throughout the method should be described in detail. Note that methods for metabolites may also be needed. Recovery data should be obtained concurrently with the residue analyses to validate the method and establish its LOQ. The experimental design of any validation studies available should be described including:

  (i) Identity of the test compounds and representative crop or commodity tested,

  (ii) Magnitude of spiking levels,

  (iii) Number of replicates per test compound per level.

- Dates of sample spiking, extraction and analysis of extracts should be listed. If extracts are not analysed on the day of preparation, storage conditions should be described.

- Raw data such as sample weights, final volumes of extracts, and peak heights/areas should be provided for control, spiked (including those for storage stability data, if required) and treated samples to support reported residue values and recoveries.

- Instrumentation should be identified, including equipment and reagents used and the operating conditions of the instrumentation. If the extraction/clean-up procedure is complex, a flow diagram should be submitted.

- Copies of representative chromatograms should be supplied for control, spiked, and treated samples along with a few sample calculations of residue levels and percent recoveries using the raw data. Examples of calibration curves of analytical standards should also be provided.

Results and Discussion

This section should contain the scientific results of the study and the relevance of results should be discussed in relation to the proposed uses of the plant protection product.

- Narrative and tables describing the steps taken in determining the pesticide residues in samples at different stages of the procedure. All graphical presentations of the data should be accompanied by the tables of the actual values from which the graphs were constructed, including processed commodity total weights for each sampling point if appropriate.

- A table of structures and chemical names/designations for the parent compound and metabolites.

- Levels of the residues (uncorrected for recovery) should be reported for each commodity analysed within the procedure (including control (untreated) samples). The individual values should be listed for all samples (not averages or ranges). If the parent pesticide and the metabolites are measured separately, the residues of each analyte should be reported. Recovery percentages (all values, not just averages or ranges) for the pesticide and/or the metabolites should be reported for all crop matrices studied.

- Dates when the samples were collected, frozen, thawed, and analysed should be provided. Storage duration and temperature of these samples should be specified. Storage stability data showing the behaviour of residues as a function of time in the relevant commodity can be referenced if available from other studies. Where samples are not analysed within the approved storage interval for the respective RAC, data should be presented showing that the storage did not affect the results of the study.

- Discussion should be presented as to whether the data indicate consistent results with the nature of residues in processed crops (high temperature hydrolysis) study, if degradation products from that study were included in the analytical phase of the processing study.

- Processing factors should be described and reported with an example calculation using unrounded residue values.
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- Deviations from the intended test protocol and the effects on the results should be described.

Conclusion

A conclusion must be reached as to whether quantifiable residues are expected following use of the pesticide at maximum seasonal application rates and timings. If residues above limits of quantification are found in the processed matrices, the results can be summarized, preferably in a table. A discussion should be provided as to the significance of the residues in the processed commodities and the distribution behaviour of the active ingredient and metabolite/degradation products, i.e., in which processed commodities and at what levels; quantifiable residues can be expected. Comparison of processing factors should also be discussed if two or more tests are conducted within one study and described in one final report.

Tables/Figures

- Tables (for example):

  (i) Name, structure, purity, for all reference standards and metabolites utilized in study.

  (ii) Distribution and quantity of parent and metabolites in various processed commodities.

  (iii) HPLC/GC retention times and TLC Rf values for active ingredient, metabolites, and related compounds under different column, solvent (elution) conditions.

- Figures (for example):

  (i) Discussion or diagram of location and size of the processing location.

  (ii) Overall extraction and fractionation strategies or schema employed for each sample matrix analysed.

(iii) Distribution of residues in processed commodities.

(iv) Flow diagrams or charts of the entire procedure.

References

Appendices

- Representative chromatograms, spectra, etc. (as applicable).

- Cite or reference reprints of published and unpublished literature, company reports, letters, analytical methodology, etc., used by the applicants (unless physically located elsewhere in the overall data report, in which case cross referencing will suffice).

- Other. Any relevant material not fitting in any of the other sections of this report should be appended.

Study Report

37. The test report should contain the following information:

- Identification of the test pesticide active ingredient (a.i.), including chemical name; common name (American National Standards Institute (ANSI), British Standards Institution (BSI), or International Standards Organization (ISO); company developmental/experimental name; and Chemical Abstracts Service (CAS) name and number and IUPAC chemical name.

- Justification for the selection of domestic or industrial processing procedure.

- Rationale for the selection of crop or commodity to be processed.

- Include a description of the test facility.

- Level of residues in the crop or commodity to be processed.

- The processed commodities sampled in the entire procedure and a rationale for their selection.

- A description of the processing procedures used.

- The sampling times (for long lasting procedures like wine production in days) for processed samples; description of the sample and number of samples/replicates.

- A rationale for the analytes determined in the study in consideration with the results of the nature of residues in processed commodity (high temperature hydrolysis) study and/or the plant and livestock studies.

- Full details pertaining to the analytical methods, including instrumentation, equipment and reagents used and the operating conditions of the instrumentation.

- A description of the preparation and handling of the samples throughout the method. Flow diagrams of extraction/clean-up procedures should be provided for complex methods.

- Analytical data for components of the residue definition in each processed commodity. Raw data such as sample weights, final volumes of extracts, and peak heights/areas should be provided for control, spiked (including those for storage stability data) and treated samples to support reported residue values and recoveries.

- Analytical responses of standards (calibration curves).

- Method validation data, recovery and method LOQ data.

- Copies of representative chromatograms should be supplied for control, spiked, and treated samples of each commodity sampled.

- Dates of sample spiking, extraction, and analysis of extracts, if extracts are not analysed on the day of preparation, include extract storage conditions.

- Freezer storage stability data (if required).

- Summary of residue data (all analytes) in processed commodities.

- Discussion of the significance of the residues in the processed commodities and the distribution behaviour of the active ingredient, i.e., in which processed commodities and at what levels; quantifiable residues can be expected.

- Conclusion as to whether quantifiable residues are expected following use of the pesticide at maximum seasonal application rates and timings. If residues above limits of quantification are found in the processed matrices, the results can be summarized, preferably in a table, including processing factors.

LITERATURE

The following documents provide additional guidance on conducting studies on the magnitude of residues in processed commodities.

(1) OECD (2008). Guidance Document for Residues in Processed Commodities, Health and Safety Publications. In preparation.

(2) Food and Agriculture Organisation of the United Nations (2002) Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed, Rome.

(3) FAO Plant Production and Protection, Report 2004 (Paper 178): Pesticide Residues in Food, Rome, Italy 2004.

(4) FAO Plant Production and Protection, Report 2005 (Paper 183): Pesticide Residues in Food, Rome, Italy 2005.

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