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Test Guidelines Programme

DRAFT NEW TEST GUIDELINE: CROP FIELD TRIAL

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Marie-Chantal HUET
Tel: +33 (0)1 45 24 79 03; Fax: +33 (0)1 44 30 61 80; Email: marie-chantal.huet@oecd.org

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This document contains a new draft Test Guideline on *Crop Field Trial* submitted to the WNT for approval.

A draft Crop Field Trial Test Guideline was circulated to member countries through the WNT and WGP (Working Group on Pesticides) on 27th November 2008 with a deadline for response of 9th January 2009. Comments were received from: Australia; Canada; the European Union including Germany, the Netherlands and the United Kingdom; New Zealand; the United States; and BIAC (Syngenta). The comments received and responses to these comments are compiled in a separate document.

ACTION REQUIRED: ***The WNT is invited to approve the draft Crop Field Trial Test Guideline, revised as appropriate.***

DRAFT CROP FIELD TRIAL TEST GUIDELINE

PURPOSE AND SCOPE

1. Crop field trials (also referred to as supervised field trials) are conducted to determine the magnitude of the pesticide residue in or on raw agricultural commodities, including feed items, and should be designed to reflect pesticide use patterns that lead to the highest possible residues. Objectives of crop field trials are to (1) quantify the expected range of residue(s) in crop commodities following treatment according to the proposed or established good agricultural practice (GAP); (2) to determine, when appropriate, the rate of decline of the residue(s) of plant protection product(s) on commodities of interest; (3) to determine residue values such as the Supervised Trial Median Residue (STMR) and Highest Residue (HR) for conducting dietary risk assessment; and (4) to derive maximum residue limits (MRLs). Crop field trials may also be useful for selecting residue definitions by providing information on the relative and absolute amounts of parent pesticide and metabolites.

2. For the purposes of this document the terms "crop field trial" and "supervised field trials" are synonymous. The term "crop field trial" will be used in the remainder of the document. In addition to addressing studies for residues in crops grown in fields (i.e., outdoors), this guideline also includes studies to assess residues in protected crops grown in greenhouses (glass or plastic covering) and in crops treated after harvest (e.g., stored grains, wax or dip treatment of fruits).

3. This Crop Field Trial test guideline provides a harmonized approach to conducting and reporting crop field trials in OECD countries. This guideline, along with the Guidance Document on Overview of Residue Chemistry Studies, provides for generation of complete field trial data sets for pesticide uses on crops in comprehensive submissions to all OECD countries.

GENERAL CONSIDERATIONS

4. A complete data set in the context of this guideline is the number of crop field trials matching the critical GAP (*c*GAP) which are required for setting an appropriate MRL and obtaining a new use of a pesticide on a crop). A reduced data set on the other hand refers to a reduced number of crop field trials matching the *c*GAP which may be adequate to obtain a new or amended registration or MRL for a plant protection product on a specific crop. A reduced data set may be sufficient where no residues are anticipated at or above the limit of quantitation (LOQ). This may be the result of a very long pre-harvest interval (PHI), or with seed treatment, pre-emergence or pre-plant uses of a plant protection product for example. This crop field trial guideline provides guidance for determining when complete data sets are necessary for determining MRLs and when it may be feasible to set an MRL using a reduced data set.

5. Bridging studies provide an essential tool in a harmonized approach to formulation changes, new formulations, and different application methods**.** A bridging study normally involves a comparison of different formulations or application methods for the purpose of data extrapolation, but may or may not involve side-by-side comparisons. If bridging trials are deemed necessary and a pesticide is used on a

wide range of crops, data should be generated for at least 3 major crop groups (one crop per crop group), e.g., a leafy crop, a root crop, a tree fruit, a cereal grain, an oilseed. The trials should be carried out on crops that would be expected to show high levels of residue (often those with applications at or near harvest). If a bridging study is conducted and residues are significantly higher with a new formulation or different application method for example, generation of a complete data set may be necessary.

6. For the special case of a comprehensive submission for a crop/pesticide combination to all OECD countries for which all crop field trials are performed at the same cGAP, a 40% reduction in total number of trials (i.e., the sum of all trials required per country or geographical region) can be achieved provided all crop field trials are submitted for evaluation and that residue levels are consistent within the whole data set. This will provide a uniform basis for exposure assessment for registration and MRL setting in all OECD countries. This provision for reduction in the number of field trials will allow all OECD regulatory authorities to gain the experience to allow them to specify broader criteria for a single international crop field trial data set.

7. Residue data from only one season are considered sufficient provided that crop field trials are located in a wide range of crop production areas such that a variety of climatic conditions is taken into account.

8. In the case of up to 25 % increases or decreases of the active ingredient application rate, the number of applications, or the PHI, under otherwise identical conditions, the residue results can be assumed to be comparable. When combining field trials for a complete data set for a crop use, this "25 % rule" may be applied to any one of the critical GAP (cGAP) components; however it is not acceptable to apply the rule to more than one *c*GAP component listed here at a time.

9. This crop field trial guideline requires one sample from treated plots at each sampling interval for crops that have 8 or more crop field trials. For seven or fewer crop field trials, some OECD countries require analysis of two independently collected samples. See paragraph 62 for more details.

PLOT AND CROP CHARACTERISTICS

Plot Size

10. Plot size may vary from crop to crop. However, plots should be large enough to allow application of the test substance in a manner which reflects or simulates routine use and such that sufficient representative sample(s) can be obtained without bias, generally at least 10 m^2 for row crops and typically 4 trees or 8 vines for orchard and vineyard crops. Plots should also be large enough to avoid contamination during mechanical sampling or harvesting if applicable. Control (untreated) plots should be located in the immediate vicinity of the treated plot(s) so that cultivation and cropping take place under similar/identical conditions. Where treated and control plots are in close proximity, measures should be taken to avoid contamination (e.g., covering or shielding crop if necessary). It is also important to ensure that plots are adequately buffered or separated. There is no minimum distance between plots which ensures adequate buffering, however prevailing wind, slope and distance between plots should all be considered prior to designing the field trial.

11. Post-harvest treatments on stored products such as potatoes, grains and seeds are often carried out in a number of storage locations with variable conditions in regard to temperature, humidity, aeration, etc. Information should be available on the use practice and all the conditions under which the treated commodities are kept. How commodities are stored during application can vary from commodities stacked in sacks, box stores and heaps to automated systems in large-scale silos or automated systems for fruit treatment.

Crop Variety

12. Crop variety may influence the uptake of the active ingredient and the metabolism capability. Residue trials should identify which crop varieties were utilized. In a set of residue trials, a selection of commercially important varieties of a crop (e.g., table and wine grapes), seasonal variations (e.g., winter wheat vs. spring wheat), vegetation period of different varieties, different maturation periods (e.g., early and late maturing fruit varieties) and morphologic variability (e.g., cherry tomatoes) should be considered. This will provide a range of conditions of use that are representative of actual agricultural situations.

Crop Maintenance and Horticultural Practices

13. Trials should be conducted in regions where the crops are predominantly grown commercially and should reflect the main types of crop maintenance and agricultural practice, especially those which can significantly impact residues (e.g., bagged and unbagged bananas, furrow and overhead irrigation, pruning of grape leaves).

Crop and Plot Maintenance Products

14. Additional plant protection measures, which are not the subject of crop field trials, are often required for crop management during the course of a study to control weeds, disease or other pests (also may include fertilizers, plant growth regulators, etc.). These crop and plot maintenance products should be chosen from among those products which do not affect (i.e., interfere with) residue analyses for the components of the relevant residue definition. Additionally, these maintenance products should be applied to both the control and treated plots in the same manner (i.e., rate and timing).

Soil Type

15. Soil type (e.g., sand, loam, sandy loam) should be identified and reported for all crop field trial sites. If the product is directly applied to soil, the field trials should include field sites with different soil types.

Greenhouse Uses

16. There are a number of protected crop scenarios such as greenhouse (glass or plastic covering), plastic tunnel, shade house, etc. which offer varying degrees of protection from environmental conditions. In matters related to residue trial conduct, greenhouse production is defined as a crop grown in its entirety (i.e., planting to harvest) in a completely enclosed structure.

TEST SUBSTANCE

Test Substance Handling

17. The test substance is the product or formulation used in a crop field trial for the purpose of generating residue data for a specific crop or commodity.

Storage

18. The test substance(s) should be stored under appropriate conditions for the study duration and applied soon after preparation or mixing.

Environmental conditions

19. Test substance applications should not be made in strong wind, during rain or when rainfall is expected shortly after application.

Active ingredients in tank-mixes, pre-mixes, sequentials

20. If residue data are generated for a single active ingredient, there are no additional data requirements for tank mix, pre-mix or other types of combinations with other active ingredients as long as there is no evidence of synergism associated with the combination(s) and as long as the *c*GAP for the active ingredient is not exceeded with any of the combinations.

21. In many cases, active ingredients may be applied in combination (i.e., tank mix, pre-mix or sequential) in crop field trials to a single treated plot as long as there is clear analytical separation (i.e., no analytical interference) of active ingredients and any relevant metabolites. A single sample may then be collected from the treated plot and prepared for residue analysis for two or more active ingredients. The exception to the combination of active ingredients in this manner would be those that are known to be synergistic, but will not be formulated together in registered products.

Formulations

22. The formulation tested in crop field trials should be as close as possible to the intended end-use product for the crop or commodity. The requirements in this guideline in regard to a complete data set (the number of crop field trials matching the *c*GAP which are required) are generally based upon only one formulation type being requested for use on a specific crop. Data needed to register additional formulation types or classes will be addressed on a case-by-case basis. In some instances a complete data set will also be needed for a new type of formulation, whereas other formulation classes may be registered with bridging studies (which normally represent a reduced data set compared to the original formulation) or possibly no additional residue data at all. The decision will be based upon how similar the formulations are in composition and physical form, the mode of application, and the timing of the application. General data requirements for additional formulation types are given in the following paragraphs.

23. Controlled release formulations (e.g., certain microencapsulated products) normally require a complete data set tailored to that particular use. Since these formulations are designed to control the release rate of the active ingredient, increased residues are possible compared to other formulation types.

24. Most of the remaining types of formulations can be divided into two groups—those which are diluted with water prior to application and those which are applied intact. Granules (GR) and dusts (DP) are the most common examples of the latter. Granular formulations applied intact will generally require a complete data set regardless of what data are already available for other formulation types. This is based on several observed cases of residue uptake being quite different for granules versus other types of formulations of the same active ingredient. No residue data will be required for dusts if data are available at the *c*GAP for a formulation of the active ingredient applied as a wetting spray (e.g., emulsifiable concentrates (EC), wettable powders (WP)).

25. The most common formulation types which are diluted in water prior to application include EC, WP, water dispersible granules (WG), suspension concentrates (SC)(also called flowable concentrates), and soluble concentrates (SL). Residue data may be translated among these formulation types for applications that are made to seeds, prior to crop emergence (i.e., pre-plant, at-plant, and pre-emergence applications) or just after crop emergence. Data may also be translated among these formulation types for applications directed to the soil, such as row middle or post-directed applications (as opposed to foliar treatments).

26. Some formulations are often designed specifically for seed treatment use such as DS powder for dry seed treatment use and ES emulsion for seed treatment. Residue data for seed treatment uses may be translated between such formulations.

27. For late season foliar applications of formulations diluted in water, the decision on the need for additional data depends upon two factors: (1) the presence of organic solvents or oils in the product and (2) the pre-harvest interval. Wider extrapolation of data will generally be permitted for formulations that do not contain organic solvents or oils (e.g., WG, WP, SC). Provided the pre-harvest interval is longer than 7 days, such formulations will be considered equivalent for residue purposes. When the PHI is less than or equal to 7 days, bridging data (see paragraph 29) will normally be needed to show residues are equivalent from these formulations. One exception to this point is that water dispersible granular formulations are sufficiently similar to wettable powders to allow translation of residue data between them regardless of the PHI.

28. Data needs for formulations containing organic solvents or oils (e.g., EC, water in oil emulsions (EO)) differ depending upon the regulatory authority. Some authorities group such formulations with those discussed in paragraph 27. Therefore, if the PHI exceeds 7 days, data may be translated between formulations such as WG, WP and EC. However, for other authorities crop field trial data for formulations such as EC or EO will normally not be translated to any other formulations unless the use is as described above in paragraph 25 (i.e.,, early season or soil applications). For mid- to late-season uses of formulations like EC or EO, these authorities would require that data as described in paragraph 29 be provided to establish whether data from another formulation can be used to support their registrations.

29. For those cases where residues are not assumed to be equivalent from two formulations, two options for bridging data are available. A reduced data set, with a 50% reduction in the number of crop field trials (and an absolute minimum of 4 trials) required for the initial formulation, may be adequate for the new formulation. In these trials, only the new formulation needs to be applied to the crop. Provided that the MRL established using the original formulation would not be increased when the data for the new and original formulations are combined, additional crop field trials for the new formulation type are not required. However, if the MRL needs to be increased based on the combined data, a complete data set will normally be required for the new formulation type. As an alternative to field trials using only the new formulation, data from a study consisting of at least 3 crop field trials with a side-by-side comparison of the two formulation types could be provided. If residues from the new formulation type are comparable to or less than those from the registered formulation, the new formulation may be considered equivalent from a residue perspective with no additional data. However, if residues are higher from the new formulation in the side-by-side study, a complete data set will be required for the new formulation type.

30. In situations where formulations are being compared for uses on numerous crops, bridging data (following either option described in the previous paragraph) are not needed for all crops provided residue similarity can be established on three major crop types, e.g., a leafy crop, a root crop, a tree fruit, a cereal grain, an oilseed, etc. Such trials should preferably be conducted on crops expected to show high levels of residues.

31. If applicants wish to register two or more formulation types which are not considered equivalent, a complete data set would typically be required for one formulation in addition to bridging studies for each additional formulation.

32. Placing a formulation (typically WP) in a water soluble bag does not require additional residue data provided adequate data are available for the unbagged product.

33. Some active ingredients (e.g., phenoxy herbicides) can be applied as one or more salts and/or esters. Different salts of an active ingredient may be considered equivalent for residue purposes in most cases regardless of the timing of the application. However, examples for which additional data may be needed for a new salt include the presence of counter ions that impart surfactant properties, significantly change the degree of dissociation, or chelate with the active ingredient ion.

34. In the case of different esters of an active ingredient, the situation is similar to that in paragraph 28 for formulations containing oils or organic solvents. Some authorities consider that different ester formulations of an active ingredient result in comparable residues when applied at PHIs longer than 7 days. If the PHI is less than or equal to 7 days, these authorities treat different esters as new formulations of that active ingredient for the purposes of determining data needs. Thus, a new ester could be subject to a reduced data set (50% fewer trials than initial formulation with absolute minimum of four trials per crop) or compared to the original ester of the active ingredient in a study with at least three trials having side-by-side plots. Other authorities require the reduced data set or side-by-side trials on a new ester for all uses other than those described in paragraph 25 (i.e., early season or soil applications).

35. Generally it is not considered necessary to provide residue data for a change in active ingredient concentration within a specific formulation type, provided the *c*GAP is not changed significantly as a result (e.g.. no more than 25% increase in amount of active ingredient per unit area).

36. Changes in formulations on the basis of a change in the content of formulants (e.g., solvents) need to be evaluated on a case by case basis. Solvents and other inert components may have an influence on the uptake or movement of the active ingredient into the plant. Special consideration should be given to changes in the content of formulants like wetting agents which may lead to better penetration of the active ingredient into the plant, particularly when the PHI is equal to or less than 7 days. In such a situation, at least a bridging study may be needed to show that residues of the active ingredient and relevant metabolites are not significantly increased by the addition of a new formulant.

Diluents and Carriers

37. Additional residue data may be required when using a diluent or carrier other than water (e.g., vegetable oil, mineral oil). The need for these data will be determined on a case-by-case basis.

Adjuvants

38. Adjuvants are products added to the spray tank for the purpose of improving the performance of the test substance or active ingredient. Adjuvants such as wetting agents, spreader-stickers, non-ionic surfactants, and crop oil concentrates may result in better deposition, penetration, or persistence of pesticide residues in or on the plant. Therefore, for a test substance which has a label allowance for the use of an unspecified adjuvant, crop field trials must include an adjuvant (any locally-available adjuvant), applied according to the label recommendation of the adjuvant. For a test substance which has a label recommendation for the use of a specific adjuvant, crop field trials must include the adjuvant, or another adjuvant with similar properties, applied according to the label recommendation of the adjuvant.

APPLICATION PARAMETERS

Spray Volume

39. Spray volumes may differ depending on the target crop or target pest (e.g., tree crops versus row crops). Crop field trials should be carried out according to the typical commercial practice(s) in regard to spray volume ensuring that the range of volumes utilized is captured. The spray volume (per unit surface

area) should be recorded in all cases. For more information on aerial applications and comparison to ground sprays, refer to paragraph 54 ("Equipment and Mode of Application").

Expression of Application Rate

Application rate

40. For all applications, the application rate should be expressed in terms of amount of product and/or active ingredient per unit area (e.g., kg a.i. per hectare or lb a.i. per acre) and where appropriate, the concentration (e.g., kg a.i./100 liters or lb a.i./100 gal) at which it is applied.

Plant height and volume

41. Row crops (potatoes, wheat, soybeans, etc.) are typically treated with broadcast sprays for which plot area (length X width) is a key consideration. In contrast, for some crops such as tree nuts, tree fruits, trellised vegetables and vines, the crop height, crown height or tree height (i.e., treated foliage height) should be recorded in order to allow crop row volume or tree row volume estimations or rate per unit area calculation as needed.

Solution concentration

42. Special consideration may be needed for foliar applications to 'tall' crops (e.g., orchard and vine crops, hops, greenhouse tomatoes), where flat boom spraying is not common practice and (air assisted) mist blowing equipment is often used. It is important to consider and report both the spray concentration (e.g., kg a.i./100 liters) and spray volumes (e.g., liters spray mixture/ha) at the various crop growth stages when planning and conducting crop field trials in these crops.

Seed treatment uses

43. Application rates for seed treatments are normally expressed as amount of active ingredient per unit of seed weight (e.g., g a.i./1000 kg seed) and seeding rate (e.g., kg seed/hectare).

Post-harvest uses

44. For dip or drench of fruit, concentration of the active ingredient in solution should be recorded ((e.g., kg a.i./100 liters (or hL)) as well as the amount of fruit treated per volume and contact time in seconds. Where dips are replenished to maintain the active ingredient concentration during treatment (i.e., where residue stripping occurs), the additional 'top-up' treatments should also be recorded. For powdering, fogging or spraying of stored goods (e.g., potatoes or grains), the application rate should be recorded (e.g., kg ai/ton or 1000 kg).

Fumigation uses

45. The application rate for gases and aerosols used in fumigation should be expressed as amount per unit volume of treated bulk good (e.g., g a.i./m^3).

Application Rate, Timing and Frequency

Maximum label rate

46. The maximum label rate or maximum proposed label rate of the active ingredient (according to the *c*GAP) should be used when applying the test substance for crop field trials.

Number of applications and re-treatment interval

47. The maximum number of applications and minimum re-treatment interval for use of the test substance under evaluation should reflect the *c*GAP.

Pre-harvest interval (PHI) in days versus final application at a specific growth stage

48. Application timing is governed by plant growth stage (e.g., pre-bloom, 50% head emergence, etc.) and/or as number of days prior to harvest. Any time that a specific PHI is indicated on the label (e.g., "Do not apply this product less than 14 days prior to harvest."), that specific PHI must be used in the crop field trials as a component of the *c*GAP, whereas the growth stage at application is of minor importance. Inversely, there are cases where the growth stage is a critical component of the GAP, (e.g., pre-emergence, at planting, pre-bloom, flag leaf or head emergence, etc.) while the PHI is of secondary importance. In these cases it is important to include as many varieties of the crop as possible in order to evaluate an appropriate range of PHIs (e.g., shorter and longer intervals from planting to maturity in the case of pre-emergence application to an annual crop). Basically in all trials both the growth stage at application (preferably as BBCH code) and PHI should be recorded.

Residue Decline Trials

49. Residue decline data are necessary for uses where the pesticide is applied when the edible portion (human food or animal feed) of the crop has formed or it is expected that residues may occur on the food or feed commodities at, or close to, the earliest harvest time. Residue decline data are used in residue evaluation for purposes such as: (1) determining if residues are higher at longer PHIs than requested; (2) estimating the half-life of the residues; (3) determining whether alteration of the PHI to levels represented in the decline trials around the GAP PHI affects the residue levels; (4) allowing for a degree of interpolation to support use patterns, including PHIs, not directly equivalent to those used in the trials on a case-by-case basis; (5) determining the profile of the residue over time to add to the understanding of metabolism of the pesticide under conditions more applicable to GAP and to assist in appropriate selection of residue definitions; and (6) determining the time interval to reach maximum residues for a systemic compound applied to crops such as potatoes or peanuts.

50. When residue decline data are necessary, some regulatory authorities require that up to 50% of the residue trials be decline studies to demonstrate the behavior of the active ingredient and relevant metabolites close to harvest.

51. When residue decline data are necessary, sampling of more than one commodity or matrix per crop may be needed. This will be the case whenever different commodities are used as food or feed at different growth stages of the crop (e.g., cereal forage, cereal fodder, cereal grain and straw). This will result in two or more sets of sampling dates within one residue decline trial.

52. The design of residue decline studies should include 3 to 5 sampling intervals in addition to the target PHI (if practical, include 0 day sampling). These sampling intervals should be spaced somewhat equally and, where possible, sampling should occur at shorter and longer time points relative to the target

PHI, when such is permitted by the window of commercial maturity. For *c*GAPs including multiple applications, a sampling point immediately prior to the final application is desirable to determine the contribution of earlier applications and the effect on residual half-life.

Reverse Decline Trials

53. Another acceptable residue decline study design option, referred to as "reverse decline," involves applications being made to separate plots at different time intervals from the targeted commercial harvest date. All plots are then harvested on the same day, the commercial harvest date, resulting in different intervals from last application to harvest. Such a design may be appropriate for situations where the commodity is likely to be harvested within a narrow time window. For example, such a study could examine the use of a pre-harvest desiccant close to maturity where harvest must occur within a short time frame after application.

Equipment and Mode of Application

Ground versus aerial application

54. Provided the proposed use does not involve ultra-low volume spraying or diluents other than water (e.g., vegetable oils), crop field trials using actual aerial application equipment can generally be waived where adequate data are available from use of ground equipment reflecting the *c*GAP as long as the product label specifies that aerial applications are to be made in spray volumes of 2 gallons or more per acre (18.7 liters or more per ha) for row crops, or 10 gallons or more per acre (93.5 liters or more per ha) for tree and orchard crops.

Hand-held versus commercial equipment

55. Application of the test substance may be made with hand-held or commercial equipment as long as the equipment is conducive to calibration procedures. Hand-held equipment used to make test substance applications in crop field trials should do so in a manner that simulates commercial practice. If single unit (e.g., one tomato) residue data need to be generated, the use of small plot precision sprayers not representative of the variability expected under commercial spraying applications should be avoided. Consideration should also be given to selection of appropriate nozzles in these trials.

Alternative application modes to the same crop

56. There are a number of soil application methods such as pre-emergence, pre-plant incorporated, in-furrow at planting, drip/drench and seed treatment. Many product labels give options for applications made prior to crop emergence, such as allowing the use to be pre-plant, at-plant, or pre-emergence. These soil-applied applications may be grouped for the purposes of determining the residue(s) resulting from the test substance application, i.e. pre-emergence applications which occur within one week after planting are considered equivalent to at-plant uses. If the label gives a choice of soil incorporation or subsequent surface application, residue data reflecting both modes of application will be required.

57. There are also a number of foliar application methods including broadcast and airblast. Field trials should reflect these multiple methods if permitted by pesticide product labels.

58. Typically, unless data from metabolism studies indicate differently, foliar application is considered the worst case compared to soil application or seed treatment and therefore would be considered to be the *c*GAP. This is especially the case if the foliar application is made when the food or feed commodity has formed and is directly exposed.

Multiple application modes to the same crop

59. It is also not uncommon to have more than one application mode of a product to the same crop within one growing season (e.g., seed treatment or pre-plant soil incorporation followed by foliar broadcast). Data from metabolism or radio-tracer studies will be helpful in determining the best approach for designing crop field trials leading to the highest residue scenario. In the absence of data indicating relative contributions to the final residue, trials reflecting the total treatment regimen may be needed, e.g., at-plant plus foliar applications.

FIELD SAMPLING

Raw Agricultural Commodity (RAC) Characteristics

60. Samples taken from field trials should be of the whole RAC as it moves in commerce. For some crops, there may be more than one RAC. For example, the RACs for field corn include the grain (seed), fodder (stover), and forage. Table 1 contains a list of the RACs derived from each crop. Some crops may be shipped without having been stripped, trimmed or washed; therefore these procedures should only be used on residue samples to the extent that these are commercial practices prior to shipment. Of course, data on trimmed or washed samples may be generated at the applicant's option for use in risk assessment.

Number of Samples per Site (Treated and Controls)

61. A minimum of one sample per treated plot per sample matrix is required to be collected and analyzed at each crop field trial site. In addition to the treated sample(s), one sample of each matrix should be collected from the control plot and analyzed for each field trial site. It is recommended, however, especially in trials where multiple samples are not taken for residue decline purposes, that a second treated sample be independently collected for each matrix at each site in case problems arise during shipping or residue analysis. Specific cases where certain regulatory authorities require two samples per treated plot are detailed in paragraph 62. Analysis of a second sample would also be useful in cases where the results at a particular site are suspicious or are inconsistent with results from other trial sites. Another factor that could promote the analysis of a second sample is the presence of high residues due to late-season foliar use (as opposed to early season use with residues <LOQ where analysis of a second sample adds very little data value).

62. As stated under Comprehensive Submissions (paragraphs 105-112), a minimum of eight field trials is required for any crop for which data are generated for all OECD countries. Some regulatory authorities require that more than one treated sample be analyzed per site when fewer than eight trials are submitted for a specific crop, including bridging studies which are used for purposes such as comparison of formulations or application methods. The specific requirements for NAFTA regulatory authorities are detailed in the following table for submissions limited to only NAFTA countries and those made to multiple OECD regions.

Study Type	**NAFTA Only Submission**	**Multiple OECD Regions**
Standard crop field trials	2 treated samples per site	1 treated sample per site (assuming minimum 8trials per crop)
Residue decline trial	1 treated sample per time point	1 treated sample per time point
Bridging studies	2 treated samples per plot	2 treated samples per plot (unless ≥8 trials per crop)

Composite versus Single Unit Samples

63. Composite samples are adequate for crop field trials. Applicants may also wish to generate replicate single unit samples from a field to aid defining unit-to-unit variation, which is needed for the purposes of acute dietary intake assessment.

Minimum Field Sample Size (Number and Weight)

64. Codex guidelines on minimum field sample sizes should be followed and are included in Table 1. A control crop sample should also be collected from each crop field trial site and for each crop commodity (e.g., cereal forage, cereal fodder, cereal grain, and straw) for analysis. Control samples of each matrix are often larger than treated samples, in order to provide the needed amount for spiking with known amounts of active ingredient (and other components of the residue definition) and to determine the calibration curves for the concurrent method validation during the analytical phase of the study.

65. For commodities not included in Table 1, applicants are advised to use the guidance on minimum field sample size for a crop part having a similar form (e.g., another seed, leafy material, root or tuber).

General Sampling Procedures

66. The sample should be representative of all portions of the crop from the field and samples should be collected without bias. Standardized procedures such as the use of the Latin squares for a forage crop, selection of tree fruits from the upper, middle, and lower levels of opposing quadrants of the tree, the use of grain triers for taking core samples of commodities in bulk quantities, and sample reduction by quartering of samples from a field are desirable. See text starting in paragraph 76 and Table 1 for crop specific sampling procedures.

67. Although samples should be collected in an unbiased fashion, whenever possible, avoid edges and ends of plots which may be influenced by turning the boom or other sprayer type on and off (ends) or where spray nozzle may be designed for spray overlap (edge effect). In cases where more than one pass is made, it may also be advisable to avoid the center of the plot to avoid the possibility of high residues from improper spray overlap.

Subsampling

68. It is acceptable to subsample large commodities (e.g., head cabbage, melons, etc.) with procedures in the field such as quartering and collecting opposing quarters. However, if analyses are planned on matrices such as pulp and peel (e.g., for dietary risk assessment refinement), the whole commodity should be shipped to the analysis lab to avoid cross contamination of peel and pulp. It is acceptable to ship these samples overnight, with coolant such as “blue ice”, to the sample preparation facility as long as they are “peeled” or “pitted”, or otherwise prepared for analyses and frozen immediately upon arrival.

Shelling and seed removal

69. Shelling, removing seeds or beans from pods, etc. is acceptable in the field provided that procedures are used which eliminate the possibility of contamination. For example, using clean implements and changing gloves between plots. In cases where commodities such as peel and pulp or stone and pulp are separated for analyses, weights must be determined for each commodity

Hand versus mechanical harvesting

70. Unless specifically directed otherwise (e.g., cotton gin byproducts), plant samples for residue analyses may be collected by hand. There is no general requirement for mechanical harvesting in crop field trials. However, in order to define a realistic residue at harvest, some mechanically harvested samples may be useful.

Washing, brushing

71. Apart from superficial cleansing, i.e., removal of any extraneous matter, no intrusive cleaning should be attempted. In the case of root crops recovered with soil, where light brushing is not sufficient to remove soil, gentle minimal rinsing under cold running water may be used. (See Detailed Sampling Procedures for additional information.)

Contamination

72. To avoid contamination, it is strongly recommended to take samples from the control plot before taking samples from the treated plot. Care should be taken to ensure that such samples are truly representative and that possible contamination or spoilage through decay is avoided.

Storage, shipping conditions and duration

73. Samples should be frozen as soon as possible following collection to avoid sample deterioration and decomposition of the residue(s). It is not advisable to allow samples to thaw once frozen; therefore shipment of frozen samples should be either by freezer truck or packed in dry ice. It is however acceptable to ship samples overnight with coolant such as "blue ice" immediately after collection provided the samples are frozen upon arrival at the laboratory or processing facility as appropriate for each matrix.

74. Normal frozen storage may not be appropriate for some pesticides (e.g., fumigants) and arrangements may be necessary for immediate residue analysis.

Form to be stored (homogenate, whole RAC)

75. Samples should be stored prior to analyses according to how the storage stability study was conducted and the analytical method for the active ingredient and relevant metabolites. For example, some methods indicate that sample homogenization must be performed on the same day as extraction. As noted in the OECD Guideline "Stability of Pesticide Residues in Stored Commodities," storage of homogenates is likely to represent a worse case (i.e., more degradation) compared to the storage of a whole commodity.

Detailed Sampling Procedures

76. Additional details regarding recommendations for the sampling of mature crops at normal harvest time, specifics on commodity sample size and the portions to be analysed are provided in Table 1.

Fruits and tree nuts

77. Circle each tree or bush and select fruit from all segments of the tree or plant, high and low, exposed and protected by foliage. For small fruits grown in a row, select fruit from both sides, avoiding the ends of the row. Select the quantity of the fruit according to its density on the tree or plant, i.e., take more from the heavily laden parts. Take both large and small fruits where appropriate, as long as all samples are marketable (except when taking immature samples for a residue decline study).

Bulb vegetables, root vegetables, tuber vegetables:

78. Take samples from all over the plot, excluding the edges of the plot and the ends of the rows to avoid edge effect. The number of sampling points depends on the sample size of the crop.

79. To provide a representative sample of the raw commodity, adhering soil may have to be removed. This may be done by brushing and, if necessary, gentle rinsing with cold running water.

80. Trim off tops according to local agricultural and/or commercial practice. Details of any trimming should be recorded. Where the tops are not used as animal feed (carrots, potatoes) or for human consumption, they should be discarded; otherwise (e.g., turnips, beets) they should be bagged separately.

Brassica vegetables, leafy vegetables, stalk and stem vegetables, legume vegetables, fruiting vegetables and fungi:

81. Take the sample from all parts of the plot, avoiding the edges and ends of rows. The number of sampling points depends on the sample size of the crop.

82. Sample items of crops such as peas or beans protected from the spray by foliage and also from parts exposed to the spray.

83. To provide a representative sample of the raw commodity, adhering soil may have to be removed. This may be done by brushing and, if necessary, gentle rinsing with cold running water.

84. For Brassica and leafy vegetables, do not trim except for the removal of obviously decomposed or withered leaves. Details of any trimming should be recorded. The fate of wrapper or outer leaves should be clearly described (i.e., included with sample or discarded in the field).

Cereals

85. If the plot is small, collect the entire yield as needed. If the plot is large but mechanical harvesting is not carried out, cut not less than twelve short lengths of row chosen from all over the plot. Cut stalks 15 cm above the ground and remove the grain from the straw.

86. Care should be taken to avoid contamination when mechanical methods are used to separate the parts of the crop. The operation is best carried out in the laboratory.

87. If the plots are harvested mechanically, take not less than twelve grab samples of grain and straw from the harvester at uniform intervals over the plot to make one bulk sample each for grain and straw.

Cereals/Legumes/Grasses/Oilseeds/Pulses - forage, hay, stover, vines, straw and other animal feed

88. Cut and/or collect these commodities according to the commercial practice. If the plots are harvested mechanically, take not less than 12 grab samples from the harvester at uniform intervals over the plot. However care should be taken to avoid contamination (e.g., harvest control prior to treated plots). For crops that are windrowed, the samples should be taken from the windrow at the time corresponding to the point when used for animal feed. In the case of cutting green plant material for the production of hay, this timing would normally be when the moisture content has decreased to the typical level for hay in commercial practice. In the case of plant material which has dried before the plant is cut (e.g., stover, straw), collect the sample after cutting and not after windrowing in the field.

Sugar cane and cane tops

89. Select whole canes from 12 areas of the plot and take short (e.g., 20 cm) sections from all parts of the length of the canes. Collect samples of green cane tops, approximately 2 kg from each plot.

Pulses, Oilseeds, Coffee, Cocoa

90. Collect samples of mature seed from at least twelve parts of the plot. Where the sample is harvested by hand, seed should normally be sent to the laboratory in the pod (except for coffee and cacao beans). When mechanical harvesting is used, only the seed should normally be supplied. Take samples from the entire plot, avoiding the edges of the plot. For coffee and cacao, circle each tree or bush and select pods or fruit from all segments of the tree or plant, high and low, exposed and protected by foliage. Select the quantity of the pods or fruit according to its density on the tree or plant, i.e., take more from the heavily laden parts.

- Cotton seed, peanuts, sesame seed, rape seed: Collect at the normal stage of harvesting.
- Sunflower seed, safflower seed: When the sampling is done by hand, collect the entire ripe heads. When sampling is done mechanically, submit only the seed to the laboratory.
- Coffee and cacao beans: Take samples in a manner reflecting common practice, i.e., sample the whole bean with its shell, but without the pod or the pulp/flesh surrounding the bean. The freshly harvested produce is not normally required.

Herbs and spices; tea leaves; hops

91. Take samples in a manner reflecting common practice. Use only those plant parts which are representative of consumption.

92. For hops select cones from all parts of the plant and from both sides of the rows, high and low, exposed and protected by foliage.

93. Take samples from the entire plot, avoiding the edges of the plot.Herbs, such as parsley and chives, and hops should be sampled fresh. As fresh hop cones are not marketed, dried cones should be produced immediately.

Stored commodities

94. Trials reflecting post-harvest treatments of stored products should be carried out over a wide range of storage facilities, and the sampling technique must be carefully chosen if valid samples are to be obtained. Procedures for taking valid samples from most commodities in storage units should reflect or simulate commercial practices. Such procedures are acceptable in sampling for pesticide residue analysis and may be used if adequate references are given. The sampling procedures are usually designed for three kinds of storage conditions as described below.

Sampling from bulk

95. Obtaining a representative sample from a (large) bulk container (e.g., cereal grains or potatoes) is difficult; if possible, samples should be taken at frequent intervals from the stream during transfer into another container. A probe sample is not representative but may be acceptable if it is possible to reach every part of the storage container; and a larger number of individual samples are taken before mixing and reducing to produce a final sample. However, it is also important for the sampling procedure to generate

samples from only that portion of the store having the highest residues. For example, pesticide residues are normally higher in the surface layer of a pile of potatoes and this should be recognised in the sampling procedure. To account for the variability of residues in these situations, at least three samples should be collected and analyzed for residues.

Sampling bagged commodities

96. Sampling of the commodity within a bag must be random. A representative sample from a large stack of bags can be obtained only if every bag is accessible. This is not always possible in practice and the alternative is to obtain a sample from a number of randomly chosen bags by probing. Since pesticide treatments are often directed to the surface of the bag, selective sampling to show the effect of the position of the bag in the stack and the penetration of the pesticide into the bag may be necessary. As with bulk containers, at least three samples should be collected and analyzed.

Sampling fruit and vegetables in packing houses

97. Where post-harvest treatments are applied to fruit and vegetables in packing houses, an adequate number of samples must be taken to determine the range of residue levels resulting from variations in the treatment process. The effects on residue levels of dip or spray concentration, temperature, duration of treatment, drying (after dip treatments) and subsequent handling may need to be considered.

98. Post-harvest treated fruit and vegetables should be kept in, or packed in, commercial containers or punnets and stored at ambient or cool-room temperature according to normal commercial practice. Day zero samples should be taken once the commodity is dried. Samples should then be drawn for analysis from the commercial containers at suitable intervals representing the time expected between treatment and subsequent marketing. The rate of disappearance or degradation of some residues depends on whether the commodity is held in a sealed or partly sealed container or is open to the air.

Table 1. Raw Agricultural Commodities and Feedstuffs Derived from Crops (compiled from the FAO Manual)

Crop	Raw Agricultural Commodity	Commodity To Be Analyzed	Field Sample Size
Citrus fruit			
e.g. Orange, Lemon, Clementine, Mandarin, Grapefruit, Tangelo, Tangerine	Fruit, whole	Whole commodity. Analyze peel and pulp separately; calculate and express the residue on the whole commodity	12 fruits from several places on 4 individual trees. If this produces a sample weight of less than 2 kg, more fruit should be taken to yield a 2 kg sample
Pomefruit			
e.g. Apple, pear, quince, crabapple	Fruit	Whole commodity after removal of stems.	12 fruits from several places on 4 individual trees. If this produces a sample weight of less than 2 kg, more fruit should be taken to yield a 2 kg sample

Stone fruit			
e.g. Apricot, nectarine, peach, plum, cherry, sweet cherry, tart (sour), mirabelle	Fruit	Whole commodity after removal of stems and stones but residue calculated and expressed on the whole fruit.	12 fruits from several places on 4 individual trees If this produces a sample weight of less than 2 kg, more fruit should be taken to yield a 2 kg sample Record weight ratio of stone and flesh
Berries			
Blackberry, raspberry (black and red), boysenberry, blueberry (= bilberry), Gooseberry Huckleberry, dewberry, elderberry, loganberry	Berry	Whole commodity after removal of caps and stems.	0.5 kg from 12 separate areas or 6 bushes
Strawberry	Berry	Whole commodity after removal of caps and stems.	1 kg from 12 different plants
Cranberry	Berry	Whole commodity after removal of caps and stems.	1 kg from 12 separate areas or bushes
Currant	Fruit	Whole commodity including stems	0.5 kg from 12 separate areas or 6 bushes
Grape (table grape; wine grape)	Fruit	Whole commodity after removal of caps and stems	12 bunches, or parts of 12 bunches, from at least 4 separate vines to give at least 1 kg
(Sub)tropical fruits with edible peel			
Date, Olive	Fruit, fresh	Whole commodity after removal of stems and stones but residue calculated and expressed on the whole fruit.	1 kg from several places on 4 trees Record weight ratio of stone and flesh.
Fig	Fruit	Whole commodity.	1 kg from several places on 4 trees
Kumquat	Fruit	Whole commodity.	12 fruits from several places on 4 individual trees or more if needed to produce a 2 kg sample
(Sub)tropical fruits with inedible peel	**Note: For all tropical or sub-tropical fruits with inedible peel, analyze peel and pulp separately; calculate and express the residue (MRL) on the whole commodity**		

Avocado, Lychee (= lithi), Mango	Fruit	Whole commodity after removal of stone but calculated on whole fruit.	12 fruits from several places on 4 individual trees. (If this produces a sample weight of less than 2 kg, more fruit should be taken to yield a 2 kg sample) Record weight ratio of stone and flesh.
Banana, Plantain	Whole fruit	Whole commodity including peel after removal of crown tissues and stalks.	24 fruits. Take two fingers each from top, middle and lowest hand of four harvestable bunches. Field residue data on both bagged and unbagged bananas should be provided.
Kiwifruit, Passion fruit, Papaya (= paw paw), Pomegranate, Guava	Fruit	Whole commodity	12 fruits from several places on 4 individual trees. If this produces a sample weight of less than 2 kg, more fruit should be taken to yield a 2 kg sample
Pineapple	Fruit	Whole commodity after removal of crown.	12 fruits
Tree nuts			
Almond	Nutmeat	Whole commodity after removal of hull and shell.	1 kg from all parts of the tree, top and bottom, exposed and covered by foliage
	Hulls	Whole commodity after removal of shell and nutmeat	1 kg
Other tree nuts (hazelnut, walnut, pecan, chestnut, pistachio)	Nutmeat	Whole commodity after removal of shell, husk or hull. Chestnuts are analyzed whole in the skin.	1 kg from all parts of the tree or bush, top and bottom, exposed and covered by foliage
Coconut	Coconut (meat and liquid combined)	Whole commodity after removal of shell. Analyze meat (= flesh) and liquid (=milk) separately; calculate and express the residue on the whole edible portion (meat and liquid).	12 nuts

Roots and tubers	**Roots or tubers may be rinsed lightly in cold running water, brushing gently with a soft brush to remove loose soil and debris, if necessary, and then dab lightly with a clean tissue paper to dry.**		
Beet, fodder (= beet), mangel, Beet, sugar,	Root Tops (leaves)	Leaves with heads are separated from the roots.	12 plants
Beet, garden (= Beetroot)	Root Tops (leaves)	Whole commodity after removal of obviously decomposed or withered leaves. Leaves are separated from the roots	12 plants
Carrot	Root	Tops are carefully cut off with a knife by cutting through the bottom of the stem at the lowest point of attachment of the outer petioles. If an annulus of root tissue is thereby severed from hollow-crown roots, the material should be recombined with the roots.	12 roots (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Cassava = tapioca	Roots	Whole commodity after removing tops. .	12-24 roots from at least 6 plants (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Celeriac	Root	Remove adhering soil	12 plants
Chicory, Salsify	Root Tops (leaves)	Whole commodity after removal of obviously decomposed or withered leaves.	12 roots (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Horseradish	Root	Whole commodity after removal of soil.	12 roots (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)

Jerusalem artichoke	Tuber	Whole commodity after removing tops.	12 tubers (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Parsnip, Rutabaga (= swede),	Root	Whole commodity after removing tops.	12 roots (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Potato, sweet potato, yam	Tuber	Whole commodity after removing tops.	12 large tubers or 24 small tubers from at least 6 plants (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Radish, Turnip	Root tops (leaves)	Whole commodity after removing tops.	12 roots (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Taro	Corm foliage	Whole commodity after removing tops	12 corm (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Bulb vegetables	**Bulb vegetables may be rinsed lightly in cold running water, brushing gently with a soft brush to remove loose soil and debris, if necessary, and then dab lightly with a clean tissue paper to dry.**		
Onion, bulb, garlic, Shallot	Bulb	Whole commodity after removal of roots (and foliage) and whatever parchment skin is easily detached.	12 bulbs from 12 plants.(the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Onion, green (= spring onions)	Whole plant, without roots	Whole vegetable after removal of roots.	24 plants (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Fruiting vegetables			
Cucumber	Fruit	Whole commodity after removal of stems.	12 fruits from 12 separate plants
Eggplant (= aubergine)	Fruit	Whole commodity after removal of stems.	12 fruits from 12 separate plants, min 1 kg (in case of small varieties)
Gherkin	Fruit	Whole commodity after removal of stems.	12 fruits from 12 separate plants (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)

Muskmelon (= melon and includes cantaloupe, casaba, crenshaw, etc., but not watermelon), Pumpkin, Watermelon, Squash, winter	Fruit	Whole commodity after removal of stems. Analyze peel and pulp separately; calculate and express the residue on the whole commodity	12 fruits from 12 separate plants
Squash, summer	Fruit	Whole commodity after removal of stems.	12 fruits from 12 plants (the sample should weigh at least 2 kg - where necessary take a larger number of fruit to produce a 2 kg sample)
Tomato, pepper, bell and non-bell (= sweet pepper and chili pepper)	Fruit	Whole commodity after removal of stems.	24 fruits from small-fruiting varieties, 12 from large fruiting varieties. From 12 plants in all cases. (The sample should weigh a minimum of 2 kg - where necessary take a larger number of items to produce a 2 kg sample.)
Okra	Fruit (pods)	Whole commodity after removal of stems.	1 kg
Brassica			
Broccoli	Flower head and stem	Analyze flower head and stems discarding leaves.	1 kg from 12 plants
Brussels sprouts	Leaf sprouts	Analyze "buttons" only.	1 kg from 12 plants. Buttons to be taken from at least two levels on each plant
Head cabbage (white cabbage; red cabbage; Savoy cabbage)	Fresh heads, with wrapper leaves	Whole commodity after removal of obviously decomposed or withered leaves.	12 plants
Cauliflower	Flower head and stem	Analyze flower head and stems discarding leaves.	12 plants
Collards	Greens	Whole commodity after removal of obviously decomposed or withered leaves.	1 kg from 12 plants

Kale	Leaves	Whole commodity after removal of obviously decomposed or withered leaves.	2 kg from 12 plants sampled from two levels on the plant
Kohlrabi	Globe without leaves	Whole commodity after removal of tops and obviously decomposed or withered leaves.	12 plants
Leafy vegetables			
Cress	Leaves and stems	Whole commodity	1 kg
Lettuce, leaf, endive/escarole/scarole	Leaves	Whole commodity after removal of obviously decomposed or withered leaves	12 plants
Lettuce, head	Fresh head, with wrapper leaves	Whole commodity after removal of obviously decomposed or withered leaves.	12 plants
Mustard greens, spinach, swiss chard	Greens (leaves)	Whole commodity after removal of obviously decomposed or withered leaves	1 kg from at least 12 plants
Watercress	Leaves and stems	Whole commodity after removal of obviously decomposed or withered leaves.	0.5 kg from at least 12 plants
Lambs' lettuce	Leaves and stems	Whole commodity	1 kg
Rape greens	Greens (leaves)	Whole commodity.	1 kg
Herbs			
Parsley	Leaves, fresh	Whole commodity after removal of obviously decomposed or withered leaves	0.5 kg fresh 0.2 kg dry
Mint (Spearmint and, Peppermint)	Tops (leaves and stems)	Whole commodity	0.5 kg fresh 0.2 kg dry
Other herbs	Leaves, fresh	Whole commodity	0.5 kg from at least 12 plants

Stem and stalk vegetables			
Artichoke, globe	Flower head	Whole commodity after removal of obviously decomposed or withered leaves.	12 flowerheads (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Asparagus, Rhubarb	Spears (stems) Asparagus stems must be washed thoroughly in cold water	Stems only.	12 sticks from 12 separate plants. (The sample should weigh a minimum of 2 kg; where necessary take a larger number of sticks to produce a 2 kg sample)
Celery	Untrimmed leaf stalk (petiole)	Whole commodity	12 plants
Leek	Whole plant	Whole commodity	12 plants, min 2 kg
Fungi			
Mushroom	Cap and stem	Whole commodity after removing adhering soil.	12 items (the sample should weigh at least 0.5 kg - where necessary take a larger number of items to produce a 0.5 kg sample)
Fresh legumes and pulses			
Bean, fresh[1]	Beans (green) with pods; Succulent (green) seeds	Whole commodity (either whole bean with pods or without depending on target crop).	Beans with pods: 24 units or 0.5-1 kg ; succulent (green) seeds: 1 kg
	Bean fodder and hay	Whole commodity	0.5-1 kg
Bean, dry [2]	Dry seeds	Whole commodity (without pods)	1 kg
	Straw	Whole commodity	0.5-1 kg straw
Cowpea	Seed	Whole commodity.	1 kg
	Hay		0.5 kg
	Forage		1.0 kg
Lentil, dry, Lupine	Seed	Whole kernel after removal of shell.	1 kg
	Fodder and straw	Whole commodity	0.5-1 kg
Mung bean[3]	Bean Bean sprouts	Whole commodity	1 kg
	Fodder	Whole commodity	0.5-1 kg

Pea, fresh	Peas (green) with pods	Whole commodity (either whole pea with pods or without depending on target crop).	24 units or min 0.5 kg
	Succulent (green) seeds		1 kg succulent (green) seeds
	Pea fodder and hay	Whole commodity	0.5-1 kg
Pea, dry	Dry seeds	Whole commodity (without pods).	1 kg dry seeds
	Vines	Whole commodity	0.5-1 kg
Pea, field[4]	Seed	Whole commodity.	1 kg
	Vines		1 kg
	Hay		0.5 kg
Cereal grains			
Barley	Grain	Whole commodity (kernel plus hull).	1 kg
	Hay	Whole commodity.	0.5 kg
	Straw	Whole commodity.	0.5 kg
Buckwheat	Grain	Whole commodity – seed plus hull	1 kg
Corn, field (= maize)	Grain	Whole commodity (grain without husk or cob)	1 kg
	Aspirated grain fractions[5]	North American requirement – Refer to OPPTS 860.1500 and Directive 98-02	
	Fodder, Stover[6]	Whole commodity	12 plants. (Cut each stem into three equal lengths (with leaves attached). Take top portion from stems 1 to 4, middle portion from stems 5 to 8 and bottom portion from stems 9 to 12, thus ensuring that parts of all 12 stems are included in the sample.)
	Forage		Forage (green or silage maize): 12 plants or min 1 kg. (Cut each stem and subsample as in previous item, retaining any cobs present on the appropriate portions of stem.)

Corn, pop	Grain	Whole commodity (grain without husk or cob)	1 kg
	Stover[6]	Whole commodity	see corn, field
Corn, sweet	Sweet corn (K + CWHR = kernels plus cob with husk removed)	Kernels plus cob without husk.	12 ears from 12 plants (the sample should weigh at least 2 kg - where necessary take a larger number of items to produce a 2 kg sample.)
	Stover[6]	Whole commodity	see corn, field
Oats, rye, millet	Forage	Whole commodity.	1 kg
	Hay		0.5 kg
	Straw		0.5 kg
	Grain		1 kg
Rice	Straw	Whole commodity.	0.5 kg
	Grain		1 kg
Sorghum	Grain	Whole commodity.	1 kg
	Forage		1 kg
	Stover[7]		1 kg
	Aspirated grain fractions[5]	North American requirement – Refer to OPPTS 860.1500 and Directive 98-02	
Sorghum, sweet	Stalk	Whole commodity	0.5 kg
Sorghum forages, Sudan grass	(See Grass)	Whole commodity	1 kg
Triticale	Grain	Whole commodity.	1 kg
	Forage		1 kg
	Hay		0.5 kg
	Straw		0.5 kg
Wheat	Grain	Whole commodity	1 kg
	Forage		1 kg
	Hay		0.5 kg
	Straw		0.5 kg
	Aspirated grain fractions[5]	North American requirement – Refer to OPPTS 860.1500 and Directive 98-02	
Oilseeds			
Rape = rape seed = oilseed rape = canola	Seed	Whole commodity.	0.5 kg
	Fodder and straw	Whole commodity	0.5-1 kg
Cotton	Undelinted seed	Whole commodity.	1 kg, with or without fibre from 12 points in the plot.
	Cotton gin byproducts[8]		0.5 kg
Flax = linseed	Seed	Whole commodity.	0.5 kg from at least 12 separate areas of each plot

Peanut	Nutmeat	Whole commodity.	1 kg
	Hay		0.5 kg
Safflower	Seed	Whole commodity.	0.5 kg
Sesame	Seed	Whole commodity	1-2 kg from 12 separate areas of plot.
Soybean	Forage	Whole commodity	1 kg
	Hay	Whole commodity	0.5 kg
	Seed, dry	Whole commodity.	0.5 kg
	aspirated grain fractions[5]	North American requirement – Refer to OPPTS 860.1500 and Directive 98-02	
Sunflower	Seed, dry	Whole commodity	0.5 kg
Seeds beverages			
Cacao bean	Bean	Whole commodity.	1 kg
Carob bean	Bean, green		1 kg
Coffee	Bean		1 kg
Others			
Ginseng	Root, dried	Whole commodity.	12 roots (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample
Hops	Hops cones, dried	Whole commodity.	Take green cone samples from at least 4 hop plants. Select cones from all parts of the plant, top and bottom, exposed and protected by foliage. Final product is at least 0.5 kg dried cones
Pimento = allspice	Fruit	Whole commodity after removal of stems.	24 fruits from small-fruiting varieties, 12 from large fruiting varieties. From 12 plants in all cases. (The sample should weigh a minimum of 2 kg - where necessary take a larger number of items to produce a 2 kg sample.)
Spices[9]	Fresh (as marketed)	Whole commodity	0.5 kg (0.2 kg dry)
Sugarcane and cane tops	Cane	Whole commodity	Min 2 kg. Select whole canes from 12 areas of the plot and take short (e.g. 20 cm) sections from all parts of the length of the canes.
Tea[10] (*Camellia sinensis*)	Plucked and dried leaves	Whole commodity.	0.2 kg dry leaves

Animal forage and fodder			
Alfalfa	Forage	Whole commodity.	1-2 kg
	Hay		0.5 kg
Clover	Forage	Whole commodity.	1 kg
	Hay		0.5 kg
Crown vetch	Forage	Whole commodity .	1 kg
	Hay		0.5 kg
Grass (pasture & range-land)	Forage	Whole commodity	1 kg
	Hay		0.5 kg
Lespedeza	Forage	Whole commodity.	1-2 kg
	Hay		0.5 kg
Sainfoin	Hay	Whole commodity	1 kg
	Forage		0.5 kg
Trefoil	Forage	Whole commodity.	1 kg
	Hay		0.5 kg
Vetch	Forage	Whole commodity.	1 kg
	Hay		0.5 kg

[1] Succulent seed without pod for beans consumed as succulent shelled beans (e.g., lima beans); succulent seed with pod for edible-podded beans (e.g., snap beans)

[2] Beans consumed as dried shelled beans

[3] Data on mung bean covers sprouts except when the product is used on the sprouts *per se*.

[4] 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 (dry matter). Field pea hay: Succulent plant cut from full bloom through pod formation. Hay should generally be field-dried to a moisture content of 10 to 20 percent.

[5] Aspirated grain fractions (previously called grain dust). Dust collected at grain elevators for environmental and safety reasons. Residue data should be provided for any post-harvest use on corn, sorghum, soybeans, or wheat. For a pre-harvest use after the reproduction stage begins and seed heads are formed, data are useful unless residues in the grain are less than the limit of quantitation of the analytical method. For a pre-harvest 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). Data needs vary among regulatory authorities.

[6] Corn stover: Mature dried stalks from which the grain or whole ear (cob + grain) has been removed; containing 80 to 85 percent DM.

[7] Sorghum stover: Mature dried stalks from which the grain has been removed; containing 80 to 85 percent DM.

[8] 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 or dirt. Cotton must be harvested by commercial equipment (stripper process) to provide an adequate representation of plant residue for the ginning process. Field trials for only the stripper type of harvesting are generally needed. Data reflecting picker cotton are not required.

[9] Spices include aromatic seeds, buds, bark, berries, pods, and roots consumed and marketed primarily in their dried form.

[10] Residue data are needed on plucked (or freshly picked) leaves and dried tea.

RESIDUE ANALYSIS

99. The analytes included in the residue definition for risk assessment and enforcement that have been previously identified in the plant metabolism studies and defined using the OECD Guidance Document on the Definition of Residue should be quantified by an appropriate analytical method (Refer to OECD Guidance Document on Pesticide Residue Analytical Methods). Method recovery validation studies should be run concurrently with the residue analyses of crop field trial samples from each individual field trial in order to provide information on the recovery levels of the test compounds from the test substrates at various fortification levels using the residue analytical methods, and to establish a validated limit of quantitation.

NUMBER OF CROP FIELD TRIALS

Combination of Data Sets for a Given Commodity

100. Individual OECD countries or political regions typically require a geographic distribution of a specified finite number of crop field trials conducted at the critical GAP to generate data for the estimation of the STMR, HR and MRL. The same practice would apply to estimation of the STMR, HR and MRL when trials conducted at the same GAP are considered from more than one country or region. Provided the GAP is comparable, the results of trials conducted in two or more countries or regions would be considered in deriving the STMR, HR and MRL for a given commodity.

101. Current guidelines in OECD countries or regions specify numbers of crop field trials based on consideration of the following factors:

1. Crop production regions, often defined or identified by the crop production practices (e.g., irrigation – beneath crop canopy vs. overhead sprinkler; planting densities of fruit trees) and the soils and climatic properties of the region.

2. Significance of the crop in a production region or country, most often determined by the production area (acres or hectares) or production quantity (tons). A crop may be considered a major or minor crop based on these factors. The production area or quantity for minor crops is not defined by all regulatory authorities.

3. Significance in the diet.

102. Having taken these factors into account, regulatory authorities in different OECD countries have each determined the minimum number of crop field trials required for registration of a use on a crop and establishment of a suitable MRL.

103. Geographic distribution of field trials within a country or region serves to ensure that data will be available for trials in key crop production areas, and a sufficient variety of horticultural practices may be represented in a crop field trial data set. Specific analyses of the influence of climate and ecology on residue levels have been performed (FAO/OECD) or are still ongoing in the US and Canada. Until these investigations are completed, the degree of importance of geographic zones remains uncertain. Preliminary results however indicate that crop production practices may have more impact on residue levels than geographic zones.

104. Although crop field trials in countries or regions must be performed up to the *c*GAP for each area, to date there are no definitive analyses that would allow trials with widely varying application rates or PHIs to be combined. However, variation of +/- 25% of PHI, application rate or number of applications is currently deemed acceptable (i.e., 25% rule).

Comprehensive Submissions

105. In the case of a comprehensive submission to all OECD countries where the desired GAP is uniform (i.e., maximum 25% deviation in one of the key parameters), a 40% reduction in the total number of trials is feasible, compared to the total number of trials determined by summation of individual country requirements. The assumption is that the number of trials specified in each crop production region reflects the economic (acreage) importance and/or dietary significance of the crop within that production region. Therefore there is no need to further consider acreage or dietary intake for a crop/commodity or to determine whether a crop is major or minor in terms of acreage, diet, or trade on a global basis for the purpose of determining a minimum number of crop field trials for a comprehensive submission.

106. The reduction in the total number of trials within any OECD country or crop production region is compensated for by the total number of crop field trials making up the comprehensive submission data set and the wider geographic distribution of these data. With this 40% reduction, regulatory authorities may receive fewer crop field trials in their specific country or region; however they will actually receive a greater number of trials in total with a more comprehensive geographical distribution. There are precedents in OECD countries and regions for this approach.

107. To qualify for this comprehensive submission approach, all crop field trials must meet the following criteria:

(1) Field trials are conducted according to the *c*GAP (within +/- 25% of the application rate, number of applications or PHI). At least 50% of the trials must be conducted at or above (within 25%) the *c*GAP. For this purpose, trials whose intended application rates match the cGAP but actual rates fall up to 10% below the cGAP (e.g., due to the normal variability in preparing spray solutions) are considered acceptable. In addition, for some authorities at least 50% of the trials need to be decline studies (see paragraphs 49-52).

(2) The trials span a range of representative crop production practices for each crop including those likely to lead to the highest residues (e.g., irrigated vs. non-irrigated, trellis vs. non-trellis production, fall-planted vs. spring-planted, etc.).

108. Any reduction in the number of crop field trials should be distributed proportionally among the crop production regions as shown in the example for a 40% reduction for barley below. A table with trial numbers for crops grown throughout OECD countries is available in the OECD Guidance Document on Overview of Residue Chemistry Studies. In the event that the number of required trials changes in any given region, the total number and reduced number should be adjusted accordingly.

Country or Region	US/CAN	EU	JP	AUS	NZ	Total
Number without reduction	24	16	2	8	4	54
Number with 40% reduction	14	10	2	5	2	33

In no case may the number of trials in a given crop production region be reduced below 2. Thus, in the example the 40% reduction does not apply in Japan and therefore the total number of trials is 33 rather than 32, which is the actual 40% reduction from 54.

109. The minimum total number of trials for any crop in a comprehensive submission is eight. In addition, the total number of trials to be conducted may not be less than the requirement for any given

individual region. For example, some crops such as dried lima beans have fewer total trials (14) than required in the EU alone (16). (For more details see OECD Guidance Document on Overview of Residue Chemistry Studies).

110. It is important to keep in mind that this comprehensive strategy would only apply to an OECD-wide submission. If, for example, the MRL submission is originally submitted to the US and Canada, the crop field trial guidelines, with respect to the number of trials, for those countries should be followed. Subsequently, if MRLs in additional OECD countries are pursued, the regulatory authorities in the additional countries should be consulted to determine what residue data are required. For example, following establishment of an MRL in the US and Canada, if an MRL for the same use is pursued in the EU, the applicant may consult with EU regulatory authorities about the possibility of using residue data from the US/Canadian data submission and performing fewer crop field trials in the EU.

111. The table of trial numbers in the OECD Guidance Document on Overview of Residue Studies addresses only outdoor crop field trials and not greenhouse (glasshouse) or post-harvest treatments. For a comprehensive submission to OECD countries, with similar critical GAPs, a minimum of 8 greenhouse trials is needed. For such greenhouse trials, geographic distribution typically is not an issue. However for active ingredients which are susceptible to photodegradation, consideration should be given to locations at different latitudes.

112. The number of post-harvest trials on a commodity should be at least four, taking into consideration the application techniques, storage facilities, and packaging materials used. As stated in paragraphs 96-97, at least three samples should be collected and analyzed in studies on bulk and bagged commodities.

GENERAL INFORMATION ON CROP GROUPS AND EXTRAPOLATION

Extrapolation and Principles of Representative Commodities

113. National authorities use targeted data sets and data extrapolation to provide sufficient data for exposure assessment or for setting MRLs for both individual major and minor crop commodities, and crop commodity groups. It provides the mechanism for extending field trial data from several (typically two or three) representative commodities to related commodities in the same crop group or subgroup. Crop grouping and the identification of representative commodities are also critical for maximizing the applicability of a targeted data set determined for representative commodities for minor uses. The representative commodity (within the group) has the following properties: (1) major in terms of production and consumption and (2) most likely to contain highest residue.

114. A number of different crop and commodity grouping systems have been developed within OECD countries to identify which commodities are likely to contain similar residues, and where group or subgroup MRLs can be considered. Characteristics of crop and commodity grouping systems are as follows:

- - All or most of the crops in a group have similar pesticide use requirements (GAP within the 25% rule). Generally this means that the registered uses (label claims) also refer to the crop group or to a substantial number of the crops within the group.
- - The expected residues in all commodities in a group are similar at harvest.

115. It may be recognized that a major crop within a crop group may not have the highest residue. From a dietary exposure standpoint, using a major crop commodity as representative of the group is

acceptable to some regulatory authorities because of the small consumption of minor commodities. However, particularly with regard to regional acute intake figures, this may not be the case.

116. Subgroups are primarily indicative of form and growth habit, and normally data for at least one commodity would be needed from each subgroup to set a group MRL. For example, citrus crops are sometimes divided into large diameter (orange, grapefruit) and small diameter (lemon, lime, mandarin) subgroups. One commodity from each subgroup (e.g., orange + mandarin) would be needed for a group MRL. Likewise, orange might be extrapolated to grapefruit (same subgroup).

117. The commodity consumed may also be reflected in the sub grouping. For example, bulb vegetables are often sub grouped thusly (1) garlic, onion, shallot and (2) spring onions. The distinction is that only the bulb on those in subgroup 1 is consumed, whereas the bulb and aerial portions of the subgroup 2 may be eaten. Different residue levels might be expected on the two sub groupings for most pesticide applications. Thus, it might be possible to extrapolate from bulb onion to garlic and shallot, but not from bulb onion to spring onion.

118. Under mutual support, trials from two related commodities showing similar residue concentrations may be considered together in order to establish MRLs for both commodities when there may be an inadequate number of trials for one or both commodities. For example, there may be 8 trials for apples and 4 trials for pears, where both are conducted under the same GAP and have similar residues. Four trials would be considered to be too few for pears, but an MRL for pears could be estimated by considering both the apple and pear trials.

119. Applicants are advised to contact individual regulatory authorities for details on their policies with regard to crop groups and extrapolation of data.

Beyond the Crop Group or Wider Extrapolation

120. Extrapolation beyond a crop group may also be possible under special circumstances. A pesticide because of its use pattern, e.g., foliar application early season before edible portions form, seed treatments, or application as a directed herbicide, or because of its properties, e.g., non-systemic and rapid degradation, will consistently yield no or low concentrations of residue (< LOQ to just above the LOQ) on a wide variety of commodities. Under such circumstances it is possible to extrapolate to establish MRLs for many commodities or crop commodity groups beyond those for which field trial data have been generated.

121. Extrapolations beyond the bounds of a crop group or subgroup may also be possible on a case-by-case basis for commodities with very similar shapes, volumes, and weights. For example, in Australia, data for apple, peach, and nectarine may be translated to persimmon, a subtropical fruit.

122. Considerations of expanded crop group MRLs would be undertaken on a case-by-case basis and would be based on the following factors:

- Use pattern
- Systemic vs. non-systemic
- Stability (degradation rate)
- Residue levels measured across several crop or commodity types

123. Determination of the sameness of the GAP must take into account not only the label instructions (rate, application method, timing, PHI) but also local agronomic practices that might impact the residue level. For example, wheat is generally grown under similar practices around the world, but grapes may be

grown under widely varying practices. For the latter, care must be taken to ascertain if the relevant GAPs are actually the same. If adequate data are available, a test of the lack of difference of the data populations would be useful.

DATA REPORTING

124. Regulatory authorities recognize there are sections in the guideline which do not apply in all cases. Therefore, applicants should exercise scientific judgment in deciding which portions are germane to a specific data submission. In particular, uses such as seed treatments and post-harvest applications will have elements which are not applicable to other types of treatments or need to be modified to address the unique characteristics of these uses. For example, soil characteristics are not applicable to post-harvest applications.

(1) Summary/introduction

(A) Study ID, Title, Author(s), Publication date, Report No., Study dates
(B) Testing Laboratory
(C) Test Guideline, including deviations
(D) Purpose of studies
(E) Description and rationale for the total number of field trials and the locations chosen (countries/regions)
(F) Results (including explanations for apparently aberrant or atypical values, discussion of geographical representation (major growing areas), seasonal variation (summer/winter, wet/dry, etc.) and representative nature of types and varieties of the raw agricultural commodity).
(G) Field procedures
(H) Analytical procedures/instrumentation
(I) Method recovery validation data
(J) Storage stability Storage period for samples should be compared to those utilized in storage stability study. In the case of measurable decline in residues, provided sufficient data points are available to construct a suitable graph, the principle of interpolation could be applied to determine the decline at any point in time and calculate a "correction factor." It should be noted here however that some regulatory agencies do not permit the use of such "correction factors."
(K) Discussion (including Quality Control measures taken; GLP compliance; statistical treatments of data; and information on the levels of the components of the residue definition in or on the RAC (specific plant parts) arising from the use of the pesticide formulated product on the test crop under specific use conditions and storage stability).
(L) Conclusions

(2) Data tables and other graphic representations.

(A) Summary map of crop field study sites (by crop)
(B) Summary tables of residue results of individual field trials
(C) Graphic representations (e.g., residue decline, figures, flowcharts, etc.)
(D) Summary tables of recovery data via the analytical methodology
(E) Summary tables of storage stability validation data
(F) Chromatograms (as applicable)

(3) Information/raw data on individual field trials (specifically, each individual field trial report should include the following information):

(A) Test substance (pesticide).
(i) Identification of the test pesticide active ingredient (a.i.), including CAS and IUPAC chemical name, common name (e.g., BSI, ISO), and company developmental or experimental name.

(ii) Identification of the pesticide formulated products used in the field trial, including trade name, type (EC, WP, G, etc.), and amount of active ingredient per gallon, pound, liter, kg, etc., and manufacturer.
(iii) Information on other relevant parameters, as pertinent, (e.g., tank mates, spray additives, carrier (encapsulating polymer, etc.)).
(iv) Other. Any and all additional information the applicant considers appropriate and relevant to provide a complete and thorough description of the test substance.

(B) Test commodity (RAC).
(i) Identification of the RAC, including type/variety.
(ii) Identification of specific crop parts harvested; used in residue analytical methodology validations; and subjected to residue analysis for a determination of the components of the residue definition.
(iii) The developmental stages, general condition (immature/mature, green/ripe, fresh/dry, etc.) and sizes of the RAC at time of pesticide application(s) and at harvestings.
(iv) Other. Any and all additional information the applicant considers appropriate and relevant to provide a complete and thorough description of the RAC.

(4) Test procedures.

(A) A detailed description of the experimental design and procedures followed in the growing of the RAC, applications of the pesticide formulated products, and harvestings of samples. The information provided, which may be presented on standardized field sheets, should include (in addition to a description of the test substance and test commodity):

(i) Trial identification number.
(ii) Cooperator (name, address), test location (e.g., state, country) and year.
(iii) Field trial lay-out (e.g., size and number of control and experimental plots; number of plants per plot/unit area, number of rows per plot, length of rows and row spacing).
(iv) Cultural treatments - farming practice (cultivation, irrigation, etc.) and cropping system.
(v) Soil characteristics (name/designation of the soil type). If application rate of the pesticide is dependent on any soil properties such as percent of organic matter, these should also be described.
(vi) Methods of application (air or ground) of the pesticide formulated products, description of the application equipment, type of application (band/broadcast, soil/foliar/ directed, ULV/concentrate/dilute, other), and calibration of pesticide application equipment, including methods and dates.
(vii) Application rates (amount of active ingredient and formulated product per acre, row, volume, etc.) and spray volumes per acre or hectare.
(viii) Number and timing of applications (total number, during dormancy, pre-plant, pre-emergence, pre-bloom, etc., between-application-intervals, and treatment-to-sampling intervals (pre-harvest intervals = PHI)).
(ix) Other pesticides applied (identity (name and type of formulated products, active ingredients), rates, dates, purpose of use, indicate whether applied separately or mixed with active ingredient of interest in trials).
(x) Climatological data (record of temperature and rainfall during the growing season from the nearest weather station, and wind speed during application).
(xi) Dates (planting/sowing/transplanting, as applicable, other significant dates in the growing of the crop (e.g., husk split for tree crops), pesticide applications, harvests).
(xii) Harvest procedures (method of harvesting (mechanical/hand, from the plant/ground/flotation, etc.), type equipment used, number/weight of samples collected per replication and number of replications per treatment level, statistical nature of sampling (e.g., fruit taken from upper,

middle, and lower portions of tree exterior and interior), sample coding (cross-referenced to sample history), etc.).

(xiii) Quality control (control measures/precautions followed to ensure the fidelity of the crop field test).

(xiv) Other. Any and all additional information the applicant considers appropriate and relevant to provide a complete and thorough description of the growing of the RAC, applications of the pesticide formulated products, and harvesting of samples.

(B) A detailed description of the handling, pre-shipping storage, and shipping procedures for harvested RAC samples. The information provided, which may be presented on a standardized form, should include (in addition to a description of the test substance and the test commodity):

(i) Sample identification (means of labeling/coding).

(ii) Conditions (temperatures, container types/sizes, sample sizes, form (e.g., whole commodity; chopped), etc.) and duration of storage before shipping.

(iii) Methods of packaging for shipment (container types/sizes, sample sizes, ambient/iced, labeling/coding, etc.)).

(iv) Means of transport from the field to the laboratory.

(v) Dates (harvest, pre-shipping storage, shipping, and receipt in the laboratory).

(vi) Quality control (control measures/precautions followed to ensure the integrity of harvested samples during handling, pre-shipping storage, and shipping operations).

(vii) Other. Any and all additional information the applicant considers appropriate and relevant to provide a complete and thorough description of the handling, pre-shipping storage, and shipping procedures for harvested samples.

(C) A detailed description of the conditions and length of storage of harvested RAC samples following their receipt in the laboratory.

(D) A detailed description of the residue analyses used in determining the components of the residue definition in field trial RAC and storage stability samples. If the specified information is provided elsewhere within the overall data submission package, it need not be reiterated here. In that case, a reference to the relevant analytical methodology would be sufficient.

(E) Method recovery validation studies should be run concurrently with the residue analyses of crop field trial samples from each individual field trial in order to provide information on the recovery levels of the test compounds from the test substrates at various fortification levels using the residue analytical methods, and to establish a validated limit of quantification. The following information specific to the method validations, which may be presented on a standardized form, should include:

(i) Experimental design: Identity of test substrates (specific plant parts) and test compounds (parent/specific metabolites). Number and magnitude of fortification levels, number of replicate samples per test compound per fortification level, sample coding, control samples, etc.

(ii) Fortification procedure: Detail the preparation of the test compounds and test substrates and the manner in which the test compounds were introduced to the test substrates.

(iii) Dates: Test sample preparation (maceration/extraction/etc.), test compounds preparation (standard solutions of known concentration), residue analyses.

(iv) Residue results: Raw data, ppm or mg/kg found uncorrected (corrected values may also be reported but the basis of correction should be explained), procedures for calculating percent recoveries, recovery levels (range), and limits of quantitation and detection.

(v) Other. Any and all additional information the applicant considers appropriate and relevant to provide a complete and thorough description of analytical methodology validation procedures.

(5) Organization of data tables and forms.

(A) Tables of residue assay data for specific plant parts analyzed. Residue levels should be reported uncorrected. Corrected values may also be presented but the procedure needs to be explained with sample calculations.
(B) Tables on residue recovery values.
(C) Graphs, as pertinent (e.g., residue decline).
(D) Forms containing field trial history information.
(E) Forms containing harvesting, shipping, storage information.
(F) Tables of weather data if unusual conditions claimed to result in aberrant residues.

(6) Trial Information

(A) Geographic Location (Trial Specific information – must be provided for all trial locations)
(i) Trial ID No (Trial Specific, unequivocal identification code (e.g., Company Internal Code)
(a) Trial Deviation (List any deviations which may impact the trial results or study conclusions)
(ii) Year (the year in which the first GLP data are collected in trial)
(iii) Country
(iv) Geographic Region (e.g., EU – N, EU – S, NAFTA 1….NAFTA 14)
(v) State/Province (e.g., Bavaria/Germany)
(vi) County
(vii) City
(viii) GPS Coordinates (Optional)
(ix) Agricultural Practice of Crop Production or GAP (Optional)
Describe the agricultural practice of producing this crop in this region
(x) Crop Grouping (Optional)
(xi) Crop
Derived from EPPO plant thesaurus, can be updated by EPPO code members. In the case of post- harvest treatment of a harvested commodity, list the crop from which the harvested commodity was derived. The same applies for seed treatment. e.g., Sweet orange is written in EPPO code as CIDSI.
(a) Crop Variety (e.g., Blood orange)
(xii) Crop Code
Codes can be obtained from www.eppo.org, utilize lowest (most detailed) level
(xiii) Soil Characterization (e.g., sandy loam, sandy clay loam, etc.)

(B) Plot (Information must be provided for all plots)
(i) Plot ID (Unequivocal Plot Identification; e.g., consecutive number). Numerical field or combination
(ii) Control Plot (yes or no)
(iii) Plot Description - Describe plot specific information: e.g., plot size or area, row spacing, plant spacing, plants/area, crop height, seeding rates, number of seeds/area, exaggerated application rate, type of protection in case of a protected crop scenario, in case of a storage protection use give type, size and volume of store, also type and size of package of stored products (e.g., bulk, paper, plastic bag) etc.
(iv) Environmental Conditions
Describe abnormal weather conditions, if applicable, soil properties, any other environmental effect that might have had an impact on the results observed in this study ; for storage protection or glasshouse application give room/glasshouse temperatures/humidities
(v) Describe crop maintenance on the plot, e.g., all procedures used in planting, maintenance, and harvest, including irrigation, application of fertilizers and other maintenance chemicals

(vi) Date of planting/sowing (for permanent crops year of planting is sufficient); in case of seed treatment give date of seed treatment and date of sowing, beginning and end of flowering, beginning and end of commercial harvest

(vii) Application

(a) Application No (1, 2, ...)

Consecutive numbers of the applications. i.e., 1st application = 1, 2nd application = 2

In the case of seed treatment, the sowing of the seeds is the first application.

(b) Growth stage (BBCH) at application, height of plants at application in case of "tall crops" (e.g., vines) and both height and crown height of plants in case of tree crops

(c) Date of Application (dd/mm/yyyy)

In case of seed treatment, state the date of sowing, in case of post-harvest dip, state the date of dip. In case of storage treatment give beginning and end of treatment together with beginning and end of ventilation

(d) Method of Application

(e) Seeding Rate (Used in conjunction with seed treatment. Using this, combined with no. seeds/ Unit, one can determine TGW (Thousand Grain Weight), etc.)

- Number of seeds/unit (no. seeds/kg, no. seeds/lb)

(f) Test Item (Pesticide(s) tested in this study)

- Description of Test Item; information regarding tested Pesticide Product, End-Use Product, formulation, treated/dressed seed, etc. used in the test item applied to the trial plot, crop, and/or the harvested commodity
- Test Item Formulation Type
- Test Item Trade Name
- Test Item Active Ingredient Code/unique identifier (e.g., Company Internal Code)
- Test Item Active ingredient name(s)
- Test Item Nominal active ingredient content (e.g., grams a.i./liter)
- Test Item actual amount active ingredient applied (e.g., grams a.i./ha); for storage protection uses: application rate (e.g., kg a.i./m^3), duration of treatment (h), duration of ventilation (h)
- Test Item actual amount active ingredient/seed if seed treatment (e.g., g a.i./100 kg seed)
- Test Item cumulative amount applied
- Adjuvant Added
  - Adjuvant Type
  - Adjuvant Name
  - Adjuvant amount in Spray Volume (%)
- Amount of water used in spray application (actual)

(viii) Sampling

(a) Sampling No.

Consecutive numbering of sampling events

(b) Sample ID – Unique sample identification code

(c) Sampling Timing: Provide any information regarding the timing of the sampling, e.g., relation to application events, days after last application, etc.

PHI – pre-harvest interval

DALA - Days after last application

Days Before Harvest

(d) Growth Stage (BBCH) at sampling

(e) Date of Sampling (dd/mm/yyyy)

(f) Sampling Information (Optional)

Description of sampling method, special remarks (e.g., cabbage was harvested according to agricultural practice, 1st set of outer leaves were removed), sample handling (e.g., samples were frozen within 24 hours)

(g) Sampled Material/Commodity (Field RAC Sample)
- Analysis Sample (Description of Analysis sample)
Field Sample may be separated into several analysis samples, e.g., whole orange may be separated into a peel sample and a pulp sample for analysis (in that case also give weights of peel and pulp), aspirated grain fractions are separated from grain.
- Analysis Sample ID
- Analysis Sample Description (Optional)
- Analyte measured
- Analyte ID.
- Extraction Date (dd/mm/yyyy)
- Actual date of extraction
- Analysis Date (dd/mm/yyyy)
- Actual date of analysis
- Method ID
- Storage Stability Factor (Use of Factor, e.g., linear, first-order, etc.)
Some regulatory authorities do not allow the use of correction factors for loss of residue in storage.
- Recovery
- Residue Level (e.g., mg/kg). Some regulatory authorites do not allow this value to be corrected for recovery and rely on the measured level of the analyte. Additionally give calculated residue if appropriate (e.g., residue xy calculated/expressed as yz or acid calculated/expressed as carboxylic ester, sum of a.i. and metabolites x and y, expressed as a.i.…)
- Number of analytical replicates

(7) Analytical Methodology: Describe basic principle of analytical method(s) and their LOQ(s), Method ID or cross-reference to relevant method template
(A) Analytical Method Information
(B) Fortification Level
(C) Recovery (%)

(8) Storage Stability: Describe longest storage interval between sampling in the field and analysis in the laboratory, and cross-reference to storage stability study, as applicable.
In case where analytes are not stable throughout the duration of the study, a correction factor might be derived from a storage stability study and applied to the residue result. Should the applicant wish to calculate such a correction factor, detailed calculations should be provided. However, it should be noted that some regulatory agencies do not allow residues to be corrected for decline in storage.

ESSENTIAL DEFINITIONS

Active ingredient(s) is the component(s) of a formulation responsible for the direct or indirect biological activity against pests or diseases, or in regulating metabolism/growth, etc. A single active ingredient may be comprised of one or more chemicals or biological entities which may differ in relative activity. A formulation may contain one or more active ingredients. (FAO Specifications)

Adjuvant refers to any product added to the spray tank for the purpose of improving the performance of the test substance/active ingredient. Adjuvants may be characterized for example as wetting agents, spreader-stickers, compatibility agents, buffering agents, de-foamers, non-ionic surfactants, crop oil concentrates, etc.

Applicant refers to a company and/or person who applies for a registration, amended registration, re-registration or MRL.

Commodity group: Commodities within a commodity group are intended to have similar residue characteristics and to be suitable for setting group MRLs. Commodity groups (e.g., pome fruits, cereal grains) within the Codex Classification for Foods and Feeds are suitable for establishing group MRLs. Note that a given crop may be included in more than one commodity group. For example, wheat is a crop, and the commodities are grain (cereal grain commodity group) and straw (straw, fodder and forage of cereal grains and grasses commodity group).

Crop Field Trial – see "**Supervised Field Trial**"; these terms are considered synonymous for purposes of this guideline.

Crop field trial site is a geographically defined address/location within a country/region/state of a field, space, greenhouse or other area in/on which a pesticide field trial is conducted. A site may consist of several *plots* (areas with defined boundaries on which a crop is grown), including control and one or more treated plots, each of which receives a specific pesticide application regimen. The trial location for a post-harvest application is defined as the location where the post-harvest treatment takes place (for example treatment room or storage location). Additionally, the trial location for a seed treatment crop field trial is defined as the location where the seed is planted or sown.

Crop Group refers to a group of crops in which the expected residues on the commodities are likely to be similar (from treatment under similar GAP) and where group or subgroup MRLs can be considered. Crop grouping is based on similarities in appearance, harvestable commodity, edible portions and/or growth habits etc.

End-use product is a product containing active ingredient(s), and usually formulant(s), that is labeled with instructions for direct pest control use or application (See also 'Product')

Extrapolation refers to a system projection of data from one system to another system. In this sense, data received from one formulation can be extrapolated to another formulation under certain circumstances. In some instances, extrapolation of field trial data obtained from one commodity are used to predict the residue behaviour of another similar commodity under described circumstances and thus proposing the same MRLs for both commodities.

Formulant is any substance or group of substances other than an *active ingredient* that is intentionally added to a pest control product to improve its physical characteristics, e.g., sprayability, solubility, spreadability and/or stability.

Good Agricultural Practice in the use of Pesticides (GAP) includes the nationally authorized safe uses of pesticides under actual conditions necessary for effective pest control. It encompasses a range of levels of pesticide applications up to the highest authorized use, applied in a manner which leaves a residue which is the smallest amount practicable.

Authorized safe uses are determined at the national level and include nationally registered or recommended uses, which take into account public and occupational health and environmental safety considerations.

Actual conditions include any stage in the production, storage, transport, distribution of food commodities and animal feed. (CAC, 1995) (FAO Manual)

Critical Good Agricultural Practice (*c*GAP) is the GAP selected to represent the worst-case use scenario within the context of national, regional, or global uses that will be producing the highest possible field residues on crop commodities. It usually includes the maximum use-rate and number of applications and the minimum re-treatment and pre-harvest intervals.

Good experimental field practice is the formalized process for designing and recording the practices used in the performance of field investigations with pesticides, and which assure the reliability and integrity of the data. See Good laboratory practice. (Draft IUPAC Glossary of Terms Related to Pesticides)

Good laboratory practice (GLP) is **t**he formalized process and conditions under which laboratory studies on pesticides are planned, performed, monitored, recorded, reported and audited. Studies performed under GLP are based on the national regulations of a country and are designed to assure the reliability and integrity of the studies and associated data. The U.S. Environmental Protection Agency GLP definition also covers field experiments (see Good experimental field practice). (after OECD, 1992) (Draft IUPAC Glossary of Terms Related to Pesticides)

Highest residue – The Highest residue (HR) level (expressed as mg/kg) in a composite sample of the edible portion of a food commodity when a pesticide has been used according to the maximum GAP conditions. The HR is estimated as the highest of the residue values (typically, one from each trial) from supervised trials conducted according to maximum GAP conditions, and includes residue components defined by the JMPR for estimation of dietary intake. (new definition) (FAO Manual)

Limit of detection (LOD) is the lowest concentration of a pesticide residue in a defined matrix where positive identification can be achieved using a specified method. (Draft IUPAC Glossary of Terms Related to Pesticides)

Limit of quantitation (LOQ) is the lowest concentration of a pesticide residue in a defined matrix where positive identification and quantitative measurement can be achieved using a specified method. (Draft IUPAC Glossary of Terms Related to Pesticides)

Maximum Residue Limit (MRL) is the maximum concentration of a residue that is legally permitted or recognized as acceptable in, or on, a food, agricultural commodity or animal feedstuff as set by Codex or a national regulatory authority. The term tolerance used in some countries is, in most instances, synonymous with MRL. It is normally expressed as mg/kg fresh weight.(after FAO, 1986) (Draft IUPAC Glossary of Terms Related to Pesticides)

The **maximum residue level** is estimated as the maximum concentration of residues (expressed as mg/kg) which may occur in a food or feed commodity following Good Agricultural Practices. The estimated maximum residue level is considered by regulatory agencies to be suitable for establishing MRLs. (FAO Manual, 2002)

Pre-harvest interval (PHI) is the time interval between the last application of a pesticide to the next normal harvest**.** (Draft IUPAC Glossary of Terms Related to Pesticides)

Post-harvest treatment refers to a pesticide application to the harvested crop, which may occur before or during storage.

Product is a formulation containing one or more active constituent(s), and possibly non-active constituent(s), which is intended for application and administration, with or without dilution before use, and which is labeled with directions for use. (Australia Sec.4 Data Requirements)

Raw agricultural commodity (RAC) means the product in or nearly in its natural state intended for sale or consumption without further processing, or for processing into food for sale to the consumer. It includes irradiated primary food commodities and products after removal of certain parts of the plant or parts of animal tissue. The term "raw agricultural commodity (RAC)" means the same as "primary food commodity". (FAO Manual)

Representative commodities are those designated commodities from which extrapolations of residue levels and resulting MRLs can be made to one or more related commodities or to an entire group of commodities ('crops').

Sample is a defined representative amount of individual raw agricultural commodity unit(s) (e.g., specific number of fruits or tubers, a set weight of grain, etc.) randomly selected from a plot which may be composited for pesticide analysis.

Seed treatment application is made to the seeds of crops prior to planting or sowing, which may occur at a seed treatment facility or in the field immediately prior to planting or sowing.

Supervised field trials are residue field trials conducted on crops, typically according to the principles of Good Laboratory Practice (GLP), in order to assess the magnitude of the residues under the conditions of the critical Good Agricultural Practice (*c*GAP).

Supervised trials median residue (STMR) is the expected residue level (expressed as mg/kg) in the edible portion of a food commodity when a pesticide has been used according to maximum Good Agricultural Practice conditions. The STMR is estimated as the median of the residue values (one from each trial) from supervised trials conducted according to maximum Good Agricultural Practice conditions. (FAO Manual).

REFERENCES – CITATIONS – LINKS

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EPA – OPPTS 860.1000 Residue Chemistry Test Guidelines and 860.1500 Crop Field Trials http://www.epa.gov/opptsfrs/publications/OPPTS_Harmonized/860_Residue_Chemistry_Test_Guidelines/Series/860-1000.pdff

http://www.epa.gov/opptsfrs/publications/OPPTS_Harmonized/860_Residue_Chemistry_Test_Guidelines/Series/860-1500.pdff

PMRA – Residue Chemistry Guidelines Section 9, Crop Field Trials, Regulatory Directive 98-02
http://www.pmra-arla.gc.ca/english/pdf/dir/dir9802b-e.pdff

EU
91/414, Appendix B, General Recommendations for the Design, Preparation and Realization of Residue Trials http://ec.europa.eu/food/plant/protection/resources/app-b.pdff

91/414, Appendix D, Guidelines on comparability, extrapolation, group tolerances and data requirements for setting MRLs http://ec.europa.eu/food/plant/protection/resources/app-d.pdff
[now revision 8]

New Zealand
Data Requirements for A Food of Feed Use Clearance Plant Compounds, 41 ACVM 06/03
http://www.nzfsa.govt.nz/acvm/publications/standards-guidelines/pc-food-clearance.pdf

Australia
Australia Residue Guideline No. 24 – Residue Trials to Obtain Permanent MRLs for Crops
December 2000

Brazil (non-OECD country included for reference only)
Sindicato Nacional da Industria de Produtos Para Defesa Agricola, Sao Paulo, December 18, 2006

Other documents:

Minimum Data Requirements for Establishing Maximum Residue Limits (MRLs) including Import Tolerances; Recommendations from the Scientific Workshop held at the Pesticides Safety Directorate, York, UK on 6-8 September 1999; Doc. 2734/SANCO/99 (prepared for the European Commission by Caroline Harris and Jeff Pim, Pesticides Safety Directorate, Mallard House, Kings Pool, 3 Peasholme Green, York, YO1 7PX, UK,on 29 September 1999)
http://ec.europa.eu/food/plant/protection/resources/min_data_en.pdff

A Survey Report to Follow-up the Development of the Concept of Minimum Data Requirements for Establishing Maximum Residue Limits (MRLs) Including Import Tolerances for Pesticides (2004)
http://www.fao.org/ag/AGP/AGPP/Pesticid/JMPR/DOWNLOAD/survey_min_data_req_mrls.pdff

Report of the OECD/FAO Zoning Project (2004)
http://www.fao.org/WAICENT/FAOINFO/AGRICULT/AGP/AGPP/Pesticid/Default.htm

OECD Guidance Document on Overview of Residue Chemistry Studies [ENV/JM/MONO(2006)32]. Environment, Health and Safety Publication, series on Testing and Assessment, No. 64; series on Pesticides, No. 32; 2006 (under revision).

OECD Guidelines for the Testing of Chemicals, TG 506: Stability of Pesticide Residues in Stored Commodities. Organisation for Economic Co-operation and Development, 16 October 2007.

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OECD Guidance Document on the Definition of Residue [ENV/JM/MONO(2006)31]. Environment, Health and Safety Publication, series on Testing and Assessment, No. 63; series on Pesticides, No. 31, 2006. (under revision).

FAO Manual 2002. Submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed. Food and Agriculture Organization of the United Nations. Rome, 2002. First edition

Growth stages of mono-and dicotyledonous plants - BBCH Monograph
http://www.bba.de/veroeff/bbch/bbcheng.pdf