

# **CROP FIELD TRIAL TEST GUIDELINE**

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## **PURPOSE/SCOPE**

1. 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 and/or commercial use patterns. Objectives of supervised field trials are to quantify/define the expected range of residue(s) in crops/commodities following treatment according to the proposed or established good agricultural practice (GAP); to determine, when appropriate, the rate of decline of the residue(s) of plant protection product(s) on commodities of interest; to determine Supervised Trial Median Residue (STMR) and Highest Residue (HR) values for conducting dietary risk assessment; and to derive maximum residue limits (MRLs)
2. This Crop Field Trial test guideline provides a harmonized approach to conducting and reporting crop field trials in OECD countries. This guideline, along with guidance from the Guidance Document on Overview of Residue Chemistry Studies provides for generation of complete field trial data sets for a crop/use in comprehensive submissions to all OECD countries/regions.

## **GENERAL CONSIDERATIONS**

- 3.. A complete data set in the context of this guideline is the number of residue supervised field trials matching the *c*GAP which are required for setting an appropriate MRL and/or obtaining a new registration or new use (i.e. plant protection product in/on a crop). A reduced data set on the other hand refers to a reduced number of residue results from supervised field trials matching the *c*GAP which may be adequate to obtain a new or amended registration and/or MRL for a plant protection product in/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.
4. Bridging studies provide an essential tool in a harmonized approach to formulation changes/new formulations. 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, 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 seed crop. The trials should be carried out on crops that would be expected to show high levels of residue (i.e. 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.

5. For the special case of a comprehensive submission for a crop/pesticide combination to all OECD countries/regions for supervised field trials performed at the same cGAP, a 40% reduction in total number of trials (i.e. the sum of all trials required per country/geographical region) can be achieved given that the whole data package is 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. Future steps for consolidation of data requirements must proceed in manageable steps. However, this provision for reduction in the number of field trials will allow the competent authorities in all OECD countries/regions to gain the experience to allow them to specify broader criteria for a single international crop field trial data set.

6. Residue data from only one season are considered sufficient provided that supervised field trials are located in a wide range of crop production areas and take into account a variety of crop production practices,

7. In the case of up to 25 % increases or decreases of the active substance 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 cGAP component listed here at a time.

8. This supervised field trial guideline requires one sample from treated plots at each sampling interval for studies that have 8 or more supervised field trials. For seven or fewer supervised field trials, some OECD countries/regions require analysis of two independently collected samples.

## **PLOT/CROP CHARACTERISTICS**

### **Plot size**

9. Plot size may vary from crop to crop. However, plots should be large enough to allow application of the end-use product 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<sup>2</sup> for row crops and typically 4 trees/8 vines for orchard/vineyard crops. Plots should also be large enough to avoid contamination during mechanical sampling/harvesting if applicable. Control (i.e. untreated) plots should be 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..

10. 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**

11. Crop variety may influence the uptake of the active ingredient and the persistence of residues. Residue trials should include data on common crop varieties. A mix 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 physiological variability (e.g. cherry tomatoes) should be considered when designing supervised field trials. This will provide a range of conditions of use that are representative of commercial situations.

### **Crop Maintenance/Horticultural practices**

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

### **Crop/Plot Maintenance products**

13. Additional plant protection products, which are not the subject of supervised 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/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, plot/crop maintenance products should be applied to both the control and treated plots in the same manner (i.e. rate and timing).

### **Soil type**

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

### **Greenhouse Uses**

15. 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.

## **APPLICATION AND REGISTRATION OF TEST SUBSTANCE**

### **Test Substance Handling**

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

#### *Storage*

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

#### *Ambient Conditions*

18. 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*

19. 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 cGAP for the active ingredient is not exceeded with any of the combinations.

20. Except in cases of synergism, test substances may be applied in combination (i.e. tank mix, pre-mix or sequential) in supervised 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 metabolites of concern. A single sample may then be collected from the treated plot and prepared for residue analysis for two or more active ingredients.

### **Formulations**

21. The formulation tested in supervised 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 residue supervised field trials matching the cGAP 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, a reduced data set 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, results of bridging studies, if appropriate, and the timing of the application. General requirements for registering additional formulation type(s) are given in the following paragraphs.:

22. Microencapsulated or controlled release formulations normally require a complete data set. Since these formulations are designed to control the release rate of the active ingredient, increased residues are possible compared to other formulation types complete data sets are needed.

23. 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 and dusts 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 cGAP for a formulation of the active ingredient applied as a wetting spray (e.g. emulsifiable concentrates (EC), wettable powder (WP)).

24. The most common formulation types which are diluted in water prior to application include EC, WP, water dispersible granules (WDG, WG) or dry flowables (DF), flowable concentrates (FIC), and soluble concentrates (liquid or solid) (SC, 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).

25. 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 preharvest interval. Wider extrapolation of data will generally be permitted for formulations that do not contain organic solvents or oils (e.g., WP, FIC, SC). Provided the preharvest interval is 7 days or longer, such formulations will be considered equivalent for residue purposes. When the PHI is less than 7 days, bridging data will normally be needed to show residues are equivalent from these formulations. One exception to this point is that dry flowable or water dispersible granular formulations are sufficiently similar to wettable powders to allow translation of residue data between them regardless of the PHI.

26. Data for formulations containing organic solvents or oils (e.g., EC's, oil dispersions) will normally not be translated to any other formulations unless the use is as described above in paragraph (c)(i.e., early season or soil applications). For mid- to late-season uses of these formulations, at a minimum bridging data would be needed to establish whether data from another formulation can be used to support their registrations.

27. For those cases where residues are not assumed to be equivalent from two formulations, two options are available. A reduced data set, with a 25% reduction in the number of supervised field trials required for the initial formulation, may be adequate for the new formulation. Alternatively, data from a bridging study consisting of at least 3 supervised 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 bridging study, a complete data set will be required for the new formulation type.

28. In situations where formulations are being compared for uses on numerous crops, bridging data 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 seed crop, etc. Such trials should preferably be conducted on crops expected to show high levels of residues.

29. 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 or a reduced data set for each additional formulation.

30. Additional information on formulations: 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.

31. Some active ingredients (e.g. phenoxy herbicides) can be applied as one or more salts and/or esters. The different salt and ester formulations of an active ingredient should result in comparable residue results when applied at PHIs longer than 7 days. While different salts of an active ingredient may be considered equivalent for residue purposes in most cases, even at shorter than 7 day PHI, different esters should be treated 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 (25% fewer trials than initial formulation) or compared to the registered form of the active ingredient in a bridging study. Examples for which additional data may be needed for a new salt include the presence of counter ions that impart surfactant properties, significantly alter dissociation constants, or chelate with the active ingredient ion.

32. The concentration of the active ingredient in a formulation is often influenced by the physical/chemical properties of that ingredient in combination with those of the other formulants. Generally it is not considered necessary to provide residue data for a change in active ingredient concentration, provided the cGAP is not changed significantly as a result (i.e. more than 25% increase in amount of active ingredient per unit area).

33. 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 lead to better penetration of the active substance into the plant, particularly when the PHI is less than 7 days. In such a situation, a bridging study at a minimum will likely be needed to show that residues of the active substance are not significantly increased by the addition of a new formulant.

### **Diluents/Carriers**

34. 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**

35. For a test substance which has a label allowance for the use of an unspecified adjuvant, supervised 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, supervised field trials must include the adjuvant, or another adjuvant with similar properties, applied according to the label recommendation of the adjuvant. Special consideration should be given to adjuvants such as wetting agents and stickers which may lead to better penetration of the active substance into the plant.

## **APPLICATION VOLUME AND RATE**

### **Spray volume**

36. Spray volumes may differ depending on the target crop and/or target pest (e.g. tree crops versus row crops). Supervised field trials should be carried out according to the typical commercial practice(s) in regard to volume. 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 Section ---..

### **Expression of application rate**

#### *Application rate*

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

#### *Plant Height/Volume*

38. Row crops (potatoes, wheat, soybeans, etc.) are typically treated with broadcast sprays (plot area = length X width). 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/tree row volume estimations or rate/unit area calculation as needed.

#### *Solution Concentration*

39. Special consideration may be needed for foliar applications to 'tall' crops (e.g. orchard and vine crops, greenhouse tomatoes), where flat boom spraying is not common practice. It is important to consider both the spray concentration (e.g. kg a.i./100 liters)

and dilute spray rates (e.g. 200-400 liters/ha) at the various crop growth stages when planning and/or conducting supervised field trials in these crops.

#### *Seed Treatment Uses*

40. Application rates for seed treatment are normally expressed as amount of active ingredient per seed weight (e.g. g a.i./1000 kg seed) and seeding rate (i.e kg of seed/hectare or acre).

#### *Post Harvest Uses*

41. For dip or drench of fruit, concentration should be recorded ((eg. 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 potatoes or grains, concentration should be recorded (eg. kg ai/ton or 1000 kg).

#### *Fumigation Uses*

42. The application rate for gases and/or aerosols used in fumigation should be expressed as amount per unit volume treated (e.g. g a.i./m<sup>3</sup>).

### **Application rate, timing and frequency**

#### *Maximum Label Rate*

43. The maximum label rate or maximum proposed label rate (according to the cGAP) should be used when applying the test substance for residue trials.

#### *Number of Applications and Re-treatment Interval*

44. The number of applications and re-treatment interval for use of the test substance under evaluation should be consistent with the cGAP. .

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

45. Application timing may be defined 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 identified and listed on the label (e.g. "Do not apply this product less than 14 days prior to harvest."), the specific PHI must be used in the supervised field trials as a component of the cGAP, and the growth stage at application is relatively less important. However, there are times when the growth stage is a critical component of the GAP, (e.g. pre-emergence, at planting, pre-bloom, flag leaf or head emergence, etc), and in such cases the PHI is relatively less important. In these cases it is important, however, to include as many varieties as possible in order to evaluate both short and long PHIs (e.g. shorter and longer intervals from planting to maturity in the case of pre-emergence application to an

annual crop). In all cases both the growth stage at application (preferably as BBCH code) and PHI should be recorded.

### **Residue decline trials**

46. Residue decline data are only necessary for uses where the pesticide is applied when the edible portion of the crop has formed or it is clear 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 to: (1) determine if residues are higher at longer PHIs than requested; (2) determine the approximate half-life of the residues; (3) decide on the range of trial PHIs acceptably close to GAP PHI; (4) allow 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; and (5) determine the profile of the residue over time to aid an understanding of metabolism of the pesticide under conditions more applicable to GAP and to assist in appropriate selection of residue definitions.

47. When residue decline data are necessary, some competent authorities require that up to 50% of the residue trials be decline studies to demonstrate the behavior of the active ingredient and other components of the residue definition close to harvest.

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

49. 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 cGAPs that include multiple applications, a sampling interval immediately prior to the final application may be important to determine the contribution of earlier applications and the effect on residual half-life.

### **Reverse Decline Trials**

50. 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 window. For example, after applying a pre-harvest desiccant close to maturity for which harvest must occur within a short time frame after application.

### **Equipment and mode of application**

*Ground versus aerial application*

51. Provided the proposed use does not involve ultra-low volume spraying or diluents other than water (e.g., vegetable oils), supervised field trials using actual aerial application equipment can generally be waived where adequate data are available from use of ground equipment reflecting the cGAP as long as the product label specifies that aerial applications are to be made in a minimum of: 2 gallons per acre (18.7 liters/ha) for row crops, or 10 gallons per acre (93.5 liters/ha) for tree and/or orchard crops. Such a design may be appropriate for situations where the commodity is likely to be harvested within a narrow window. For example, after applying a preharvest desiccant close to maturity and harvest must occur within a short time frame after application.

*Hand-held versus commercial equipment*

52. 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 supervised field trials should do so in a manner that simulates commercial practice and ensures uniformity.

*Alternative application modes to the same crop*

53. 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 preplant, at-plant, or preemergence. These soil-applied applications may be grouped for the purposes of determining the residue(s) resulting from the test substance application, i.e. preemergence applications which occur within one week after planting are considered at-plant. If the label gives a choice of soil incorporation and/or subsequent surface application, residue data reflecting both modes of application will be required.

54. 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.

55. Typically, unless data from metabolism studies indicate differently, foliar application is considered the worst case compared to soil application and therefore would be considered to be the cGAP.

*Multiple application modes to the same crop*

56. 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/radio-tracer studies will be helpful in determining the best approach for designing supervised 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.

**FIELD SAMPLING**

### **Raw Agricultural Commodity (RAC) Characteristics**

57. 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 seed, fodder, 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.

### **Sampling Procedures**

58. 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. For general sampling procedures, see Table 1.

### **Number of Samples per Site (treated and controls)**

59. A minimum of one sample per treated plot per sample matrix is required to be collected and analyzed at each supervised field trial site. In addition to the treated sample(s), one control sample of each matrix should be collected and analyzed for each field trial site. It is strongly 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. Analysis of the second sample would be useful in cases where the results at a particular site are suspicious or are inconsistent with results from other trial sites. Other factors that could promote the analysis of a second sample include the presence of high residues due to late-season foliar use (as opposed to early season use with residues <LOQ) and when more data are needed for risk assessment purposes.

60. As stated under Comprehensive Submissions in Section ---, 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.

### **Composite versus Single Unit Samples**

61. Composite samples are adequate for supervised residue trials. Where applicants may wish to generate replicate samples for risk assessment, the variation between replicate field single unit samples from a trial may be used as an aid in defining unit-to-unit variation, where the unit-to-unit variation information is needed for the purposes of acute dietary intake assessment.

### **Minimum sample size (number and weight)**

62. Codex guidelines on minimum 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.

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

### **Sampling: and sample handling**

#### *Random Collection*

64. Samples should always be collected randomly (i.e. without bias). 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*

65. 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 contamination. 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*

66. Shelling, removing seeds/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/or changing gloves between plots/treatments. 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*

67. Unless specifically directed otherwise (e.g. cotton gin byproducts/gin trash), plant samples for residue analyses may be collected by hand. There is no general requirement for mechanical harvesting in supervised field residue trials.

### *Washing, brushing*

68. 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*

69. To avoid contamination, it is strongly recommended to take samples from the control plot before taking samples from the treated plot. In order to define a realistic residue at harvest, some mechanical harvested samples may be useful. Care should be taken to ensure that such subsamples are truly representative and that possible contamination or spoilage through decay is avoided.

70. Another aspect of avoiding contamination is 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 be considered.

### *Storage, shipping conditions and duration*

71. 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. (refer to OECD Storage Stability Guideline)

### *Form to be stored (macerate, whole RAC)*

72. Samples should be stored prior to analyses according to the analytical method for the test substance and relevant metabolites. For example, some methods indicate that sample homogenization must be performed on the same day as extraction.

## **Detailed sampling procedures**

73. 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*

74. 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:*

75. 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.

76. 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.

77. 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:*

78. 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.

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

80. 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.

81. Do not trim except for the removal of obviously decomposed or withered leaves. Details of any trimming should be recorded.

*Cereals*

84. 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. Crops which are harvested mechanically can be sampled from the harvester as it proceeds through the crop, however care should be taken to avoid contamination (e.g. harvest control prior to treated plots). For crops that are windrowed, collect the fodder after cutting and not after windrowing in the field.

*Sugar cane and cane tops*

91. 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*

93. 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. When mechanical harvesting is used, only the seed should normally be supplied. Take samples from the entire plot, avoiding the edges of the plot.

– 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*

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

95. 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.

96. Take samples from the entire plot, avoiding the edges of the plot.

97. Herbs, such as parsley and chives, should be sampled fresh. In the case of hops, both fresh and dried cones should be collected.

## *Stored Commodities*

98. Supervised trials of 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*

99. Obtaining a representative sample from a (large) bulk container (e.g. of 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. Pesticide residues are normally higher in the dust fraction and this should be recognised in the sampling procedure.

### *Sampling bagged commodities*

100. 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.

### *Sampling fruit and vegetables in packing houses*

101. 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/spray concentration, temperature, duration of treatment, drying (after dip treatments) and subsequent handling may need to be considered.

102. 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	Commodity Sample Size
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<b>Crop</b>	<b>Raw Agricultural Commodity</b>	<b>Commodity To Be Analyzed</b>	<b>Commodity Sample Size</b>
<b>Citrus fruit</b>			
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
<b>Pomefruit</b>			
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
<b>Stone fruit</b>			
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
<b>Berries</b>			
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
<b>(Sub)tropical fruits with edible peel</b>			
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.

Crop	Raw Agricultural Commodity	Commodity To Be Analyzed	Commodity Sample Size
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
<b>(Sub)tropical fruits with inedible peel</b>	<b>Note: For all tropical/sub-tropical fruits with inedible peel, analyze peel and pulp separately; calculate and express the residue (MRL) on the whole commodity</b>		
Avocado, Guava, Lychee (= lithi), Mango, Pomegranate	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)	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
<b>Tree nuts</b>			
Almond	Nutmeat	Whole commodity after removal of shell.	1 kg from all parts of the tree, top and bottom, exposed and covered by foliage
	Hulls	Whole commodity after removal of 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)	12 nuts

Crop	Raw Agricultural Commodity	Commodity To Be Analyzed	Commodity Sample Size
		separately; calculate and express the residue on the whole edible portion (meat and liquid).	
<b>Roots and tubers</b>	<b>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.</b>		
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

Crop	Raw Agricultural Commodity	Commodity To Be Analyzed	Commodity Sample Size
			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 very 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)
<b>Bulb vegetables</b>	<b>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.</b>		
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, w/o 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)
<b>Fruiting vegetables</b>			
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	Fruit	Whole commodity after removal of stems.	12 fruits from 12 separate plants

<b>Crop</b>	<b>Raw Agricultural Commodity</b>	<b>Commodity To Be Analyzed</b>	<b>Commodity Sample Size</b>
cantaloupe, casaba, crenshaw, etc., but not watermelon), pumpkin, watermelon		Analyze peel and pulp separately; calculate and express the residue on the whole commodity	
Squash  = summer winter	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-fruited varieties, 12 from large fruited 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
<b>Brassica</b>			
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, w/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
<b>Leafy vegetables</b>			
Cress	Leaves and stems	Whole commodity	1 kg

<b>Crop</b>	<b>Raw Agricultural Commodity</b>	<b>Commodity To Be Analyzed</b>	<b>Commodity Sample Size</b>
Lettuce, leaf, endive/escarole/scarole	Leaves	Whole commodity after removal of obviously decomposed or withered leaves	12 plants
Lettuce, head	Fresh head, w/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
<b>Herbs</b>			
Herbs	Leaves, fresh	Whole commodity.	0.5 kg from at least 12 plants
Parsley	Leaves, fresh	Whole commodity after removal of obviously decomposed or withered leaves	0.5 kg fresh 0.2 kg dry
Mint (Spearmint/Peppermint)	Tops (leaves and stems)	Whole commodity	0.5 kg fresh 0.2 kg dry
<b>Stem and stalk vegetables</b>			
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
<b>Fungi</b>			
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)

Crop	Raw Agricultural Commodity	Commodity To Be Analyzed	Commodity Sample Size
<b>Fresh legumes and pulses</b>			
Bean, fresh <sup>1</sup> Bean fodder and hay	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, dry <sup>2</sup>	Dry seeds	Whole commodity (without pods)	1 kg
	Straw	Whole commodity	0.5-1 kg straw
Cowpea <sup>3</sup>	Seed	Whole commodity.	1 kg
	Hay		0.5 kg
	Forage		1.0 kg
Lentil, dry, Lupin Fodder and straw	Seed	Whole kernel after removal of shell.	1 kg
Mung bean <sup>4</sup> Fodder	Bean bean sprouts	Whole commodity	1 kg
Pea, fresh Pea fodder and hay	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, dry	Dry seeds	Whole commodity (without pods).	1 kg dry seeds
	Vines	Whole commodity	0.5-1 kg
Pea, field <sup>5</sup>	Seed	Whole commodity.	1 kg
	Vines		1 kg
	Hay		0.5 kg
<b>Cereal grains</b>			
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 <sup>6</sup>	North American requirement – Refer to OPPTS 860.1500	
	Fodder, Stover <sup>7</sup>	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.)

<b>Crop</b>	<b>Raw Agricultural Commodity</b>	<b>Commodity To Be Analyzed</b>	<b>Commodity Sample Size</b>
	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 <sup>7</sup>	Whole commodity	see corn, field
Corn, sweet	Sweet corn (K + CWHR)	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 <sup>7</sup>	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 <sup>7</sup>		1 kg
	Aspirated grain fractions <sup>6</sup>	North American requirement – Refer to OPPTS 860.150	
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 <sup>6</sup>	North American requirement – Refer to OPPTS 860.1500	
<b>Oilseeds</b>			
Rape = rape seed = oilseed rape = canola Fodder and straw	Seed	Whole commodity.	0.5 kg
Cotton	Undelinted seed	Whole commodity.	1 kg, with or without fibre from 12 points in the plot.

<b>Crop</b>	<b>Raw Agricultural Commodity</b>	<b>Commodity To Be Analyzed</b>	<b>Commodity Sample Size</b>
	cotton gin byproducts <sup>9</sup>		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		1 kg
	Hay		0.5 kg
	Seed, dry	Whole commodity.	0.5 kg
	aspirated grain fractions <sup>6</sup>	North American requirement – Refer to OPPTS 860.1500	
Sunflower	Seed, dry	Whole commodity	0.5 kg
<b>Seeds beverages</b>			
Cacao bean	Bean	Whole commodity.	1 kg
Carob bean	Bean, green		1 kg
Coffee	Bean		1 kg
<b>Others</b>			
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-fruited varieties, 12 from large fruited 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 <sup>10</sup>	Fresh	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 <sup>11</sup>	Plucked and dried leaves	Whole commodity.	0.2 kg dry leaves

Crop	Raw Agricultural Commodity	Commodity To Be Analyzed	Commodity Sample Size
<b>Animal forage/fodder</b>			
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

<sup>1</sup> 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)

<sup>2</sup> Beans consumed as dried shelled beans

<sup>3</sup> Cowpea is the only bean crop considered for livestock feeding (see cowpea). Residue data for forage and hay are required only for cowpea.

<sup>4</sup> Data on mung bean covers sprouts except when the product is used on the sprouts per se.

<sup>5</sup> 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.

<sup>6</sup> Aspirated grain fractions (previously called grain dust). Dust collected at grain elevators for environmental and safety reasons. Residue data may be provided for any postharvest use on corn, sorghum, soybeans, or wheat). For a preharvest use after the reproduction stage begins and seed heads are formed, data are useful unless residues in the grain are less than the limit of quantitation of the analytical method. For a preharvest use during the vegetative stage (before the reproduction stage begins), data will not normally be needed unless the plant metabolism or processing study shows a concentration of residues of regulatory concern in an outer seed coat (e.g. wheat bran, soybean hulls). Data needs vary among national/regional regulatory authorities.

<sup>7</sup> Corn stover: Mature dried stalks from which the grain or whole ear (cob + grain) has been removed; containing 80 to 85 percent DM.

<sup>8</sup> Mature dried stalks from which the grain has been removed; containing 80 to 85 percent DM.

<sup>9</sup> Cotton gin byproducts (commonly called gin trash). Include the plant residues from ginning cotton, and consist of burrs, leaves, stems, lint, immature seeds, and sand and/or dirt. Cotton must be harvested by commercial equipment (stripper 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.

<sup>10</sup> Spices include aromatic seeds, buds, bark, berries, pods, and roots consumed and marketed primarily in their dried form.

<sup>11</sup> Residue data are needed on plucked (or freshly picked) leaves, dried tea, and instant tea.

## **RESIDUE ANALYSIS**

103. 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 **OECD Guidance on Establishing Residue Definitions for Pesticide Residue Limit/Tolerance Setting** should be quantified by an appropriate analytical method (Refer to **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 SUPERVISED FIELD TRIALS**

### **Combination of Data Sets for a Given Commodity**

104. Individual OECD countries or political regions typically require a geographic distribution of a specified finite number of supervised field trials conducted at the critical GAP to generate field trial 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 political region. Provided the GAP is comparable, the results of trials conducted in two or more countries/political regions would be considered in deriving the STMR, HR and MRL for a given commodity.

105. Current guidelines in OECD countries/regions specify number 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/or the soils and climatic properties of the region.

(2) Significance of the crop in a production region and/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.

Having taken these factors into account, regulatory authorities in different OECD nations/regions have each determined the minimum number of supervised field trials required for authorization and to establish a suitable MRL.

106. Geographic distribution of field trials within a country/crop production 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/ecology on residue levels have been performed (FAO/OECD) or are still ongoing in US and Canada (US/CAN). 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.

107. Although supervised field trials in countries/crop production 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**

108. 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/commodity within that crop production region.

109. 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 and/or trade on a global basis for the purpose of determining a minimum number of supervised field trials where regulators may accept data sets from other countries provided comparability of *c*GAP can be demonstrated..

110. The reduction in the total number of trials within any OECD country of crop production region is compensated for by the total number of supervised 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 supervised field trials in their specific country/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.

111. To qualify for this comprehensive submission approach, all Supervised 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. In addition, for some authorities at least 50% of the trials need to be decline studies.
- (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/non-irrigated, trellis/non-trellis production, fall-planted/spring-planted, etc).

112. Any reduction in the number of supervised 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/ Region	US/CAN	EU	JP	AUS	NZ	Total
Number w/out reduction	24	16	2	8	4	58
Number w/ 40% reduction	14	10	2	5	2	37

113. 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). 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 is 33 rather than 32, which is the actual 40% reduction from 54.

114. 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 US and Canada, the crop field trial guidelines, with respect to the number of trials, for that region should be followed. Subsequently, if MRLs/authorizations in additional OECD countries/regions are pursued, the regulatory authority in the additional OECD countries/regions should be consulted to determine what residue data are required. For example, following establishment of an MRL/authorization in US/CAN, if an MRL/authorization for the same product is pursued in EU, the applicant may consult with EU regulatory authorities about the possibility of using residue data from the US/CAN data submission and performing fewer supervised field trials in EU.

115. The table of trial numbers in the OECD Guidance Document on Overview of Residue Studies addresses only supervised field trials and not greenhouse (glasshouse) or post harvest treatments. For a comprehensive submission for OECD countries, with similar critical GAPs, a minimum of 8 greenhouse trials are needed. For such greenhouse trials, geographic distribution typically is not an issue. However for products/test substances which are susceptible to photodegradation, consideration should be given to locations in both the Northern and Southern hemispheres

116. 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.

## **GENERAL INFORMATION ON CROP GROUPS/EXTRAPOLATION**

### **Extrapolation and principles of representative commodities**

117. 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 crops, and crop groups. It provides the mechanism for extending field trial data from several (typically two or three) representative crops to related crops in the same crop group or subgroup. Crop grouping and the identification of representative commodities is also critical for maximizing the applicability of a targeted data set determined for representative crops 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. (See OECD Guidance Document on Overview of Residue Studies.)

118. A number of different crop/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/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 authorised uses (label claims) also refer to the crop group.
- The expected residues in all commodities in a group are similar at harvest.

119. Competent authorities recognize 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 because of the small consumption of minor commodities.

120. 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).

121. 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) chives, spring onion, and leeks. The distinction is that only the bulb on those in subgroup 1 are 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/or shallot, but not from bulb onion to spring onion.

122. Extrapolations beyond the bounds of a crop group or subgroup may be possible in special circumstances. Consideration on a case-by-case basis may be given to 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.

123. Wider/ cross group extrapolations may also be possible for: (1) situations where residues are expected to be <LOQ (e.g., pre-emergence herbicide uses, pre-flower

treatments); (2) seed treatments; (3) post harvest treatments for non-systemic pesticides (similar size and morphology).

124. Under mutual support, trials from two related crops may be considered together in order to establish MRLs for both crops when there may be an inadequate number of trials for one or both crops. 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 residue concentrations. 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.

### **Beyond the Crop Group or Wider Extrapolation**

125. 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 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 10 X LOQ) on a wide variety of crops. Under such circumstances it is possible to extrapolate to establish MRLs for many crops or crop groups beyond those for which field trial data have been generated.

126. 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 types

127. 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**

The data which have to be reported in the full study report are supposed to reflect the structure of the residue trials template. This structure has been made up in order to fulfill the needs of structured submission of data in xml format for further data base input.

### **1. Generic part**

- 1.1. Study period
- 1.2. Full report of the study including full report of field part and analytical part
- 1.3. Applicant's Summary and Conclusion
- 1.4. Reliability and deficiencies of the study, if appropriate

### **2. Trial information (to be reported for each single trial in the study)**

#### **2.1. Geographic Location**

- 2.1.1. Trial ID No.
- 2.1.2. Indicate indoor/outdoor/storage protection use
- 2.1.3. Year of main growing season (e.g. the year after sewing if winter wheat is concerned)
- 2.1.4. Geographical location: City, County, State, Province, Postal Code, Country, Geographical Region or Zone, GPS coordinates if appropriate; include rationale for the selection of the test sites if appropriate
- 2.1.5. In case of "protected crop scenarios": kind of protection used (e.g. glasshouse, plastic tunnel, shadehouse...), in case of storage protection use: kind, size and volume of store
- 2.1.6. Agricultural Practice of Crop Production
- 2.1.7. Crop, Crop Group, Crop Code (according to EPPO), Crop variety
- 2.1.8. Soil type
- 2.1.9. Description of the general climatic conditions including Min/Max temperature and rainfall data at each test site for the duration of the study, detailed temperature and rainfall monitoring data (give source of data) around the date(s) of application; for storage protection or glasshouse application give room/glasshouse temperatures

#### **2.2. Plot information (to be repeated for every single plot)**

- 2.2.1. Plot ID
- 2.2.2. Control plot (yes/no)
- 2.2.3. Plot description: Map of the test plot, indicating its location, topography and size, and location and size of the control plot in relation to the test plot
- 2.2.4. Description of all the procedures used in planting, maintenance, and harvest, including irrigation, application of fertilizers and other maintenance chemicals
- 2.2.5. Crown height if appropriate (orchard trees)
- 2.2.6. Storage protection: kind and size of package
- 2.2.7. Date of seed treatment if appropriate
- 2.2.8. Begin and end of commercial harvest
- 2.2.9. Begin and end of flowering
- 2.2.10. Date of planting/sewing (if appropriate, for permanent crops year of planting might be sufficient)

- 2.2.11. Number and interval of applications
- 2.2.12. Application (to be repeated for every single application)**
  - 2.2.12.1. Growth stage (BBCH code) at application
  - 2.2.12.2. Height of plants at application in case of “tall crops” (e.g. vines)
  - 2.2.12.3. Date of Application; begin and end of treatment together with begin and end of ventilation in case of storage treatment
  - 2.2.12.4. Method of Application (e.g. low volume spraying, granular application)
  - 2.2.12.5. Equipment used for application
  - 2.2.12.6. Additional adjuvants/surfactants/mixing partners used for the application, usually expressed as percent (%), v/v, wt/wt
  - 2.2.12.7. Test item
    - 2.2.12.7.1. Description of test item
    - 2.2.12.7.2. Formulation used in the trial (identity, company developmental code, trade name), substance type (e.g. pure a.i., technical product, formulation), formulation type (e.g. SC, WG), **information on active substance(s) (to be repeated for each active substance in the formulation)**, which should include: code/unique identifier of the substance (chemical name; common name, Chemical Abstracts Service (CAS) name and number, IUPAC chemical name), molecular formula, molecular weight, analytical purity nominal (and actual) content of a.i. in formulation (e.g. 0.1 kg/L or 10 % v/v; 100 g/kg or 10 % wt/wt)
    - 2.2.12.7.3. Applied nominal (and actual) amount of test substance (kg a.i./ha or lb a.i./A); for storage protection: application rate (kg a.i./m<sup>3</sup>), duration of treatment (h), duration of ventilation (h); for seed treatment: seeding rate (seed units per ha, kg seed/ha) number of seeds per seed unit, kg a.i. per 100 kg seed
    - 2.2.12.7.4. Spray volume if appropriate: applied amount of water used in spray application (L/ha)
    - 2.2.12.7.5. Spray concentration if appropriate (kg a.i./hL)
- 2.2.13. Sampling (to be repeated for every single sample)**
  - 2.2.13.1. Sampling ID
  - 2.2.13.2. Growth stage (BBCH code) at sampling
  - 2.2.13.3. PHI (days), DALA, storage protection: withholding period (days)
  - 2.2.13.4. Date of sampling
  - 2.2.13.5. Further information on sampling including sampling procedure, sample weight and preparation after harvesting such as shipment, conditions and period of storage and any preparation that was done prior to extraction
  - 2.2.13.6. Description of the sampled material (commodity, matrix)
  - 2.2.13.7. Analysis sample (to be repeated for every analysis sample)**
    - 2.2.13.7.1. Analysis sample ID
    - 2.2.13.7.2. Description of the analyzed material (commodity, matrix), weight of analysis sample
    - 2.2.13.7.3. Extraction date
    - 2.2.13.7.4. Analysis date

**2.2.13.7.5. Analyte measured (to be repeated for all analytes measured in the analysis sample)**

- 2.2.13.7.5.1. Analyte measured (a.i., metabolite...), analyte ID
- 2.2.13.7.5.2. Residue of the analyte(s) (mg/kg) as non-corrected values (based on the measured analyte), calculated residue if appropriate (could be 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....; would be depending on the residue definition and the analytical method)
- 2.2.13.7.5.3. Number of analytical replicates
- 2.2.13.7.5.4. Representative chromatograms including peak heights/areas
- 2.2.13.7.5.5. Method of Analysis (Method ID)
- 2.2.13.7.5.6. Reference to the analytical methods document(s) if appropriate or brief description of the analytical method, which should then include: information on extraction and detection (instrumentation and reagents used, extract volumes, operating conditions of the instrumentation), flow diagrams of extraction/clean-up procedures should be provided for complex methods, provide comment on the analytical method's suitability, provide information on the method validation (spiking levels, range of recoveries, average recovery and standard deviation), analytical responses of standards (calibration curves), detector linearity, method LOQ, dates of sample fortification, extraction and analysis of extracts if extracts are not analyzed on the day of preparation
- 2.2.13.7.5.7. Freezer storage stability for analyte in days/months, fortification level in the storage stability study, note whether or not residues of the chemical have been shown to be stable for the duration of storage that occurred during the study

## 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.

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

**Extrapolation group/Crop Group** refers to a group of crops in which the expected residues at harvest could be similar, based on similarities in appearance, harvestable commodity, edible portions and/or growth habits etc.,

**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 (cGAP)** 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 the 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 (cGAP).

**Supervised 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 supervised field trial is defined as the location where the seed is planted or sown.

**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**

### **US and Canada**

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.pdf](http://www.epa.gov/opptsfrs/publications/OPPTS_Harmonized/860_Residue_Chemistry_Test_Guidelines/Series/860-1000.pdf)

[http://www.epa.gov/opptsfrs/publications/OPPTS\\_Harmonized/860\\_Residue\\_Chemistry\\_Test\\_Guidelines/Series/860-1500.pdf](http://www.epa.gov/opptsfrs/publications/OPPTS_Harmonized/860_Residue_Chemistry_Test_Guidelines/Series/860-1500.pdf)

PMRA – Residue Chemistry Guidelines Section 9, Crop Field Trials, Regulatory Directive 98-02

<http://www.pmr-arla.gc.ca/english/pdf/dir/dir9802b-e.pdf>

### **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.pdf>

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.pdf>

[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.pdf](http://ec.europa.eu/food/plant/protection/resources/min_data_en.pdf)

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.pdf](http://www.fao.org/ag/AGP/AGPP/Pesticid/JMPR/DOWNLOAD/survey_min_data_req_mrls.pdf)

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. Organisation for Economic Co-operation and Development. ENV/JM/MONO(2006)32. 10 October 2006.

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