Health Canada Santé Canada

Canada

Pest Management Regulatory Agency

# Updated Residue Chemistry Guidelines

**PMRA Guidance Document**

*Protecting the health and environment of Canadians*

*Protéger la santé des Canadiens et l'environnement*

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# Document history (revision/update)

| Updated | Update/Rationale: |
|---|---|
| 19 October 2022 | This Guidance Document replaces Regulatory Directive DIR98-02, *Residue Chemistry Guidelines* |

## Foreword

Guidance documents are meant to provide assistance to industry and other stakeholders on how to comply with governing science policies. Guidance documents also provide assistance to staff on how Health Canada mandates and objectives should be implemented in a manner that is consistent and effective.

Guidance documents are administrative instruments that allow for flexibility in approach. Alternate approaches to the principles and practices described in this document may be acceptable provided they are supported by adequate scientifically-based justification. Alternate approaches should be discussed in advance with the PMRA to avoid the possible finding that applicable requirements have not been met.

Health Canada reserves the right to request information or material, or define conditions not specifically described in this document. Health Canada is committed to ensuring that such requests are justifiable and that decisions are clearly documented.

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# In summary

The Health Canada Pest Management Regulatory Agency has updated the Residue Chemistry Guidelines to reflect current practices. These guidelines were first made available to registrants and applicants of pest control products in June 1998. The updated guidelines are harmonized with those established by the Organisation for Economic Co-operation and Development (OECD), various elements of which have been adopted and implemented over the years. The PMRA continues to collaborate with OECD countries in the development of test guidelines and guidance documents.

Applicants are requested to utilize residue chemistry guidelines and guidance documents in preparing, conducting, and reporting experimental studies to address the residue chemistry data requirements. These data will elucidate the nature and magnitude of residues in treated foods for purposes of dietary risk assessment and setting Maximum Residue Limits (MRLs). Data waiver requests will be considered when supported by a scientifically defensible rationale.

The harmonization of the PMRA and the OECD test guidelines will continue to greatly facilitate joint reviews and work sharing with international partners.

A phase-in period will be permitted to provide applicants with the time necessary to perform studies as per the updated residue chemistry guidelines and, hence, move towards more consistent data requirements. Studies initiated after 31 December 2023, should be in conformance with these guidelines.

In each study report, the applicant must indicate whether these guidelines have been followed. An explanation must be provided along with details on any deviation.

Applicants are encouraged to refer to the OECD webpage for any updates that may have been issued after the publication of this document.

## 1.0 Introduction

This Guidance Document, which replaces the Regulatory Directive DIR98-02, *Residue Chemistry Guidelines* published in 1998, reflects the work that the Health Canada's PMRA has completed through the OECD Residue Chemistry Expert Group (RCEG), the North American Free Trade Agreement (NAFTA), the International Crop Grouping Consulting Committee (ICGCC) and as part of the Regulatory Cooperation Council's (RCC) initiative.

The residue chemistry guidelines are intended to provide guidance on the residue chemistry data requirements that must be addressed to support an application for the registration of conventional pest control products for use on human food and animal feedstuffs in Canada or to support the establishment of a maximum residue limit (MRL) for residues in/on an imported food. The residue chemistry guidelines also provide criteria and protocols for the design, performance, validation, and reporting of scientific studies. These guidelines have been harmonized with the OECD Guidelines for the Testing of

Chemicals and OECD Guidance Documents, which were developed over many years, and were adopted by all OECD member countries, including Canada.

The objective of the OECD Test Guidelines and OECD Guidance Documents for pesticide residue chemistry is for use in identifying pesticide residues in human food or animal feedstuffs for purposes of dietary risk assessment and setting MRLs. These OECD Guidelines and Guidance Documents were developed based on guidelines previously in use in OECD member countries and by the Food and Agriculture Organization (FAO).

Applicants are requested to utilize residue chemistry guidelines and guidance documents in preparing, conducting, and reporting experimental studies to address the residue chemistry data requirements. In turn, these data allow for the elucidation of the nature and magnitude of residues in treated foods. International joint reviews and work sharing will continue to be greatly facilitated as a result of the harmonization of the OECD Test Guidelines and Guidance Documents.

It is important to note that applicants are encouraged to consult with the PMRA prior to initiating studies that may deviate from these guidelines or guidance documents.

Revisions to this document will be undertaken as scientific knowledge, evaluation, assessment tools, and/or risk management strategies evolve. Such changes, however, will be made to strengthen guidance and reduce regulatory burden.

**Reference**

OECD (2013). Introduction to OECD Test Guidelines on Pesticide Residues Chemistry - Section 5 Part A, OECD Publishing, Paris. https://doi.org/10.1787/9789264203761-en

## 2.0 Evaluation templates

Evaluation templates are used to review the scientific studies that are submitted to support applications to register pest control products or to address data call-in for re-evaluation. These templates capture specific data components and record the PMRA reviewer's conclusions and rationales that are based on each data set. Information from the individual review templates are used to create an overall review document, also known as the dietary evaluation integrated assessment, presenting the conclusions and recommendations on which the regulatory decision is based.

Health Canada's PMRA encourages registrants and applicants to complete the updated Residue Chemistry review templates for all residue chemistry studies and submit them with the application package. These templates will help PMRA scientists in their review of applications and reduce the time required to review the data package. The use of templates allows for consistency in the content, format, and facilitates the evaluation process. In addition, these templates will:

- increase efficiency and transparency;
- be an effective means of sharing information internationally, especially in joint international reviews of pesticide applications; and
- result in resource savings that will benefit the PMRA.

The residue chemistry review templates describe the layout and scope of information that should be contained within each template. Applicants may include additional information beyond that prescribed in a particular review template; however, it should be done without affecting or changing the format and content of the template. The study review templates are available for the following studies:

- Plant Metabolism
- Livestock Metabolism
- Residue Analytical Methods
- Freezer Storage Stability in Plants
- Freezer Storage Stability in Livestock
- Livestock Feeding
- Crop Residue Trials
- Processing
- Confined Crop Rotation
- Field Accumulation

These templates can be found on the United States Environmental Protection Agency's website.

Once completed, the review templates must be submitted to the PMRA under DACO Parts 12.7.6 and 12.7.7 *Applicant Generated Study Reviews*.

**References**

Health Canada (2016). Evaluation Templates. https://www.canada.ca/en/health-canada/services/consumer-product-safety/pesticides-pest-management/registrants-applicants/product-application/evaluation-templates.html

USEPA. Study Profile Templates. https://www.epa.gov/pesticide-registration/study-profile-templates

# 3.0 Use-site categories and DACO tables

Data elements required to support an application to register or amend a registration, request a MRL on imported food, conduct research with a pest control product, or address data call-in for re-evaluation depend on the nature and specific uses of the product as well as the purpose of the application.

As a guide for determining data requirements, possible use-sites have been grouped into a series of use-site categories. Each use-site category has a list of required and conditionally required data called data-code (DACO) which can be found in DACO tables.

The residue chemistry data requirements are listed under DACO Part 6 (for Technical Grade Active Ingredient and End-Use Product) and DACO Part 7 (for End-Use Product).

See PMRA's *Guidance for developing datasets for conventional pest control product applications* for detailed guidance on each listed DACO under Parts 6 and 7. An applicant can use this guidance to better understand the data required for completing their application to register a pesticide in Canada. This guidance must be used along with the use-site categories and DACO Tables information available on the Pesticides section of the Canada.ca website. The DACO tables outline the required data elements for a specific use-site category and the guidance document clarifies the conditions for each required data element.

Human food and animal feedstuff uses are part of the following use-site categories:

- Use-site Category 1 Aquaculture and Aquatic Food Sites
- Use-site Category 5 Food Crops Grown Indoors in Greenhouses or other Enclosed Structures
- Use-site Category 8 Terrestrial Animals for Food Production
- Use-site Category 10 Seed and Plant Propagation Materials: Food and Feed
- Use-site Category 12 Food and Feed Processing and Storage
- Use-site Category 13 Terrestrial Feed Crops
- Use-site Category 14 Terrestrial Food Crops
- Use-site Category 20 Structures (Food *Handling Establishments*)

**References**

Health Canada (2013). Use-Site Category (USC) Definitions for Conventional Chemical Pesticides. https://www.canada.ca/en/health-canada/services/consumer-product-safety/pesticides-pest-management/registrants-applicants/product-application/use-site-category-daco-tables/definitions-conventional-chemical-pesticides.html

Health Canada (2016). Use Site Category (DACO Tables). https://www.canada.ca/en/health-canada/services/consumer-product-safety/pesticides-pest-management/registrants-applicants/product-application/use-site-category-daco-tables.html

Health Canada (2018). Guidance for developing datasets for conventional pest control product applications: data codes for parts 1, 2, 3, 4, 5, 6, 7 and 10. https://www.canada.ca/en/health-canada/services/consumer-product-safety/reports-publications/pesticides-pest-management/policies-guidelines/guidance-developing-applications-data-codes-parts-1-2-3-4-5-6-7-10.html

# 4.0 Overview of residue chemistry studies

In 2003, the OECD initiated work to develop harmonized Test Guidelines and Guidance Documents on pesticide residue chemistry. Harmonized guidelines are essential to further align science for pesticide registration and re-registration. These harmonized guidelines were based on guidelines used in Australia, Canada, Japan, the United States, the European Union and by the Food and Agriculture Organization (FAO) for the determination of pesticide residues in human food or animal feedstuffs. Data derived from such guidelines are not only used by industry to fulfil pesticide registration requirements in countries/regions but also support FAO's development of recommendations on MRLs. The following Test Guidelines and Guidance documents have been developed by the OECD:

**Test guidelines**

- TG 501: Metabolism in Crops
- TG 502: Metabolism in Rotational Crops
- TG 503: Metabolism in Livestock
- TG 504: Residues in Rotational Crops (Limited Field Studies)
- TG 505: Residues in Livestock
- TG 506: Stability of Pesticide Residues in Stored Commodities
- TG 507: Nature of Pesticide Residues in Processed Commodities – High Temperature Hydrolysis
- TG 508: Magnitude of Pesticide Residues in Processed Commodities
- TG 509: Crop Field Trial

**Guidance documents**

- Definition of Residue
- Overview of Residue Chemistry Studies
- Residues in Rotational Crops
- Pesticide Residue Analytical Methods
- Crop Field Trials
- Magnitude of Pesticide Residues in Processed Commodities
- OECD Maximum Residue Limit Calculator

For more information, refer to OECD *Guidance Document on Overview of Residue Chemistry Studies* (as revised in 2009).

**Reference**

OECD (2009). Guidance Document on Overview of Residue Chemistry Studies (as revised in 2009), Series on Pesticides, No. 32; Series on Testing and Assessment, No. 64. http://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2009)31&doclanguage=en

# 5.0 Chemical identity

Chemical identity information is required to accurately identify the chemical structure, chemical name and the physical and chemical properties of the technical grade active ingredient (also referred to as test substance in the OECD Test Guidelines) and to elucidate its behaviour in soil, water, plants and animals, including melting point, water solubility, solvent solubilities, vapour pressure and octanol/water partition coefficient.

## Chemical pest control products

Data requirements for chemical identity are essentially the same as those discussed in *Guidance for developing datasets for conventional pest control product applications: data codes for parts 1, 2, 3, 4, 5, 6, 7 and 10*, concerning chemistry data requirements for a technical grade active ingredient and an end-use product.

If an impurity is identified in the technical grade active ingredient, and is likely to occur as a significant residue in food/feed, then residue data for the impurity may be required. The determination of whether residue data for an impurity are needed will be based on the impurity's stability, toxicity, and detectability.

## Information required for other types of agrichemicals

**Adjuvants**

Adjuvants, either present in the formulation or as stand-alone products to be added as tank-mix partners, should be fully described, including the chemical name as well as any trade names. Chemical abstracts services (CAS) registry numbers should be included, if available. The chemical names should be in the same form as those for adjuvants as described in the PMRA Regulatory Directive, DIR93-15, *Registration Requirements for Adjuvant Products*. If only the trade name is known, the applicant should request that the supplier of the adjuvant provide the descriptive information, including CAS name, structure, and purity, directly to the PMRA. For more details, refer to Section 19. Maximum Residue Limits.

The PMRA recommends conducting field trials in the presence of adjuvants as there is the possibility that the addition of an adjuvant to a pesticide spray mixture could result in higher residues of the pesticide on the crop. Crop field trials are to be carried out with the pesticide/adjuvant mixture according to the proposed use pattern in representative growing regions for the crop(s) of interest. The pesticide residues are quantified but not the adjuvant.

For a request to register a "new" adjuvant to be used with a registered end-use product which currently allows the use of a "similar-type" of adjuvant (for example, non-ionic surfactant, crop oil concentrate), the residue data for the combination pesticide-adjuvant/crop must have been previously reviewed by the PMRA when setting the MRL for the pesticide/crop combinations. Therefore, additional residue chemistry data would not likely be required unless product chemistry and/or toxicology concerns were identified for the specific adjuvant being proposed for registration. In turn, as it is not anticipated that the magnitude of the residues of the pesticide would increase by replacing a non-ionic surfactant with another non-ionic surfactant using the same use pattern, additional residue trials would not be required if the "new" adjuvant is considered acceptable with respect to the product chemistry, toxicology and efficacy/crop tolerance.

**Safeners**

As per PMRA Regulatory Directive, DIR2006-02, *Formulants Policy and Implementation Guidance Document*, a safener is defined as a formulant, in some herbicidal and other types of pest control products, that mitigates the effects of the product on specific economically important crops.

Prior to the *Pest Control Products Act*, which was enacted 28 June 2006, safeners were not included in the definition of a pest control product. Under the new section 2(1)a) of the *Pest Control Products Act* and the new Pest Control Product Regulations, this is no longer a limitation. As per section 2(b) of the new Pest Control Product Regulations:

"2. For the purpose of paragraph (c) of the definition "pest control product" in subsection 2(1) of the Act, the following are prescribed to be pest control products:

a) a device that is manufactured, represented, distributed or used to directly or indirectly control, destroy, attract or repel a pest or to mitigate or prevent the injurious, noxious or troublesome effects of a pest; and
b) a compound or substance that is not an ingredient of a pest control product described in paragraph (a) of that definition but is added to or used with such a product to enhance or modify its physical or chemical characteristics or to modify an effect on host organisms in connection with which the product is intended to be used."

Therefore, safeners must now be registered as pest control products if they are not contained as an ingredient in an end-use product. Safeners as an ingredient in an end-use product still do not require separate registration, but as safeners are biologically active, they are still subject to the same data requirements to those required for an active ingredient.

For a new safener, the residue chemistry data requirements are very similar to those of a new active ingredient for a use under use-site categories 13 and 14. However, as the safener data requirements include studies done on the safener itself (plant and livestock metabolism) as well as those conducted using the end-use product with which the safener is mixed (either as an ingredient or tank-mix), the required DACO Parts 6 and 7 must be fulfilled.

Note: The plant metabolism studies must be conducted with the herbicide/safener mixture and both the herbicide and the safener metabolic pathways need to be investigated.

Determination of residues (both pesticide and safener) in representative samples of crops (both food and feed matrices) treated with the pesticide/safener mixture must be carried out. Furthermore, determination of residues must be carried out on meat, milk and eggs if the pesticide/safener mixture is used to treat feed items.

MRLs to cover residues of the safener (and possible metabolites/degradates) are necessary. As such, an enforcement method for the determination of the safener residues is required (for both plant and livestock matrices, if applicable).

**References**

Health Canada (1993). Regulatory Directive DIR93-15, Registration Requirements for Adjuvant Products. https://www.canada.ca/content/dam/hc-sc/migration/hc-sc/cps-spc/alt_formats/pacrb-dgapcr/pdf/pubs/pest/pol-guide/dir/dir9315-eng.pdf

Health Canada (2006). Regulatory Directive DIR2006-02, Formulants Policy and Implementation Guidance Document. https://www.canada.ca/content/dam/hc-sc/migration/hc-sc/cps-spc/alt_formats/pacrb-dgapcr/pdf/pubs/pest/pol-guide/dir/dir2006-02-eng.pdf

Health Canada (2018). Guidance for developing datasets for conventional pest control product applications: data codes for parts 1, 2, 3, 4, 5, 6, 7 and 10. https://www.canada.ca/en/health-canada/services/consumer-product-safety/reports-publications/pesticides-pest-management/policies-guidelines/guidance-developing-applications-data-codes-parts-1-2-3-4-5-6-7-10.html

# 6.0 Metabolism in livestock and crops

Metabolism is also referred to as "Nature of the Residue". The purpose of conducting metabolism studies is to determine the qualitative and quantitative metabolic fate, uptake, translocation, and disposition of the active ingredient, in other words, examine what happens to the active ingredient when it is applied to a plant (directly or indirectly), to the soil (preplant or preemergence), to the water (to control invasive weeds in a lake, in paddy rice, or aquaculture), to irrigation water, or administered to livestock (through treated feedstuff or direct application). Many pesticides undergo transformations, in other words, biotic and abiotic metabolism or degradation, during or after application to the soil/growing media, water, crop, seed, or livestock. The nature, in other words, composition, of the terminal residue must therefore be determined before complete residue detection methodology and quantitative residue data can be developed. To obtain this information, the pesticide is labelled with a radioactive atom(s), to follow the translocation/disposition of the compound and determine the qualitative/quantitative profile of the active ingredient and the metabolites within a plant or animal.

Metabolism studies are required whenever a pesticide use is determined to be a human food or feedstuff use. Based on the results of the characterization/identification studies, the residue definition for (i) dietary risk assessment and (ii) setting and enforcing MRLs should be proposed by the applicant and confirmed by the PMRA's scientists (refer to Section 8 for more details). Residue analytical methods must be developed for all components of the residue definition.

The active ingredient should be labelled, to allow for residue quantitation. The preferred radioisotope is $^{14}C$, although $^{32}P$ and $^{35}S$ can be used. The identification of the components of the terminal residue and the residue definition often present complex problems that must be resolved before finalizing the analytical methodology and gathering the quantitative residue data.

The desired goal of a metabolism study is the identification and characterization of at least 90% of the TRR in edible tissues, milk, eggs and in each raw agricultural commodity (RAC) of the treated crop. In many cases, it may not be possible to identify significant portions of the total radioactive residues (TRRs) especially when low total amounts of residue are present, when incorporated into biomolecules, or when the active ingredient is extensively metabolized to numerous low level components. The determination of whether the TRRs have been sufficiently characterized/identified will depend on the level of radioactivity remaining uncharacterized/unidentified, the importance of the plant or livestock commodity with regards to dietary intake containing the uncharacterized/unidentified residue as a food or feed, the chemical structure of the active ingredient and identified metabolites, and the toxicity of chemicals similar in structure to potential metabolites. Evidence must be provided that the applicant has made every attempt to release the bound radioactivity and subsequently characterize/identify the released radioactivity. Thus, the applicant may wish to consult with the PMRA's chemists and toxicologists to determine whether the residues have been sufficiently characterized/identified, which metabolites should be covered by the MRLs and included in the risk assessment, and which components of the TRRs must be determined by the residue analytical methodology.

Studies must utilize state-of-the-art techniques and include citations of such techniques when used. Refer to Table 1 of OECD TG 501, TG 502, and TG 503 for guidance on the strategy for identification and characterization of extractable residues in crops and livestock. The applicant must delineate, preferably in a flowchart, the routes of degradation or metabolism in plants and animals, and clearly specify the capability of the analytical method(s) to determine the components of the residue definition, in the RAC sample. Photographs or autoradiographs of thin layer chromatographic (TLC) plates, radioautograms, or output from other appropriate imaging systems that were critical to the identification should be provided. Such evidence will contribute significantly to the evaluation of the data. CAS and International Union of Pure and Applied Chemistry (IUPAC) names should also be provided, in a table format, for all structures identified in the profile.

The applicant should always be aware of the possibility of new metabolites of the pesticide that may affect future MRL proposals. Where the structure of a metabolite or

transformation product is identical to another registered pesticide, the applicant should also state this fact.

The metabolism studies should also result in elucidation of the efficiency of extraction of the various components of the residue definition (also known as radiovalidation of the enforcement method) so that extraction/residue release, in other words, solubilization, procedures can be developed as part of the analytical methods (see Section 9, *Residue Analytical Methods)*.

## 6.1 Metabolism in livestock

Metabolism (also known as Nature of the Residue) in livestock studies are used to determine the qualitative and quantitative metabolism and/or degradation of the active ingredient resulting from pesticide use in animal feedstuffs (refer to Appendix I of this document for a list of feed items), direct application to livestock, or animal premise treatment. The studies provide an estimate of total residues in the edible livestock commodities, as well as in the excreta; identify the major components of the terminal residue in the edible tissues; elucidate a metabolic pathway for the pesticide in ruminants and poultry; and provide evidence whether or not residues should be classified as fat soluble.

For oral treatment, the results from the poultry metabolism study are extrapolated to all fowl, and the ruminant metabolism results are generally extrapolated to all other animals. A swine metabolism study may be required if the metabolic profile observed in the ruminant and/or poultry is significantly different from the profile observed in the rat/laboratory animal. For dermal application, a metabolism study is conducted for each target specie (for example, cattle, sheep) and extrapolation of the results to other animals is not generally considered appropriate.

**Metabolism in fish**

When fish or shellfish may be exposed to the pesticide or its degradation products, a fish metabolism study is required (For more details see Section 16, *Residues in Water, Fish, and Irrigated Crops*).

**Test guideline**

For further information, refer to OECD Test Guideline No. 503, *Metabolism in Livestock.*

**Use-site categories and DACO**

Metabolism in livestock studies are required for use-site categories 1, 8 and 13 and conditionally required under use-site categories 10, 12 and 14. These studies must be submitted to the PMRA under DACO Part 6.2.

Metabolism in livestock is not required for use-site category 5, as there are no feed items associated with greenhouse uses.

## 6.2 Metabolism in crops

Metabolism (also known as Nature of the Residue) in crops studies are used to determine the qualitative and quantitative metabolism of the active ingredient, to elucidate the degradation pathway and require the identification and/or characterization of the metabolism and/or degradation products when a pesticide is applied to a crop directly or indirectly.

The term, metabolism in crops, is used here for convenience to describe the formation of all transformation products of the pesticide in or on crops, regardless of whether they result from crop metabolic processes or degradation processes (for example, photolysis, hydrolysis).

As per OECD TG 501, crops can be considered to belong to one of five categories for crop metabolism studies: root vegetables, leafy crops, fruits, pulses and oilseeds, and cereals (refer to Annex 1 of OECD TG 501 for details). In order to extrapolate metabolism of a pesticide to all crops/crop groups, metabolism studies on a minimum of three representative crops (from the five different categories) must be conducted for a given use pattern and show similar metabolism (metabolic pathways and major metabolites). In addition, a metabolism study is necessary for genetically-modified crops that involve the insertion of a gene conveying resistance by means of metabolism.

**Test guideline**

For further information, refer to OECD Test Guideline No. 501, *Metabolism in Crops*.

**Use-site categories and DACO**

Metabolism in crops studies are required for use-site categories 1, 5, 10, 12, 13 and 14, and must be submitted to the PMRA under DACO Part 6.3.

## 6.3 Metabolism in rotational crops

Metabolism in rotational crops (also known as Confined Crop Rotation) studies are conditionally required for uses of pesticides on terrestrial food and/or feed crops. A rotational crop (also known as secondary crop) is defined as any crop which may be planted after the harvest of a pesticide-treated primary crop or replanted after failure of the pesticide-treated primary crop. Rotational crop studies are not required for uses of pesticides on asparagus, berries, cranberry, ginseng, globe artichoke, grape, mushroom, pome fruits, rhubarb, stone fruits and tree nuts.

Metabolism in rotational crops studies (which are considered Tier 1 studies) are conducted to determine the nature and amount of pesticide residue uptake in rotational crops that are used as human food or as livestock feed. The studies provide an estimate of TRRs in the various raw agricultural commodities (RACs); identify the major components of the TRRs in the various RACs; elucidate the degradation pathway of the active ingredient in rotated crops; provide data to determine appropriate rotational intervals (also known as plantback intervals; PBIs) and/or rotational crop restrictions based on

residue uptake levels; and provide information for determining if limited field trials for rotational crops (which are considered Tier 2 studies) must be performed (see Section 13, *Residues in Rotational Crops (Limited Field Studies)*, for more details). The study should be performed using a sandy loam soil treated with the radiolabelled test substance at a rate equivalent to the maximum seasonal rate (1×) among all proposed/registered uses. Three appropriate rotational intervals must be used: 7–30 days to simulate crop failure, 60–270 days to reflect a typical rotation after harvest of the primary crop, and 270–365 days for crops rotated the following year. Three rotational crops must be used and be representative of each of the following crop groupings: root and tuber vegetable, small cereal grain, and leafy vegetable. Soybeans may be substituted for a leafy vegetable due to the importance of this crop in certain rotational practices. The three rotational crops should be harvested and the appropriate RACs for human and livestock feed plant parts should be sampled and the TRR determined at all three rotational intervals.

**Test guideline and guidance document**

For further information, refer to OECD Test Guideline No. 502, *Metabolism in Rotational Crops*, and OECD Guidance Document on *Residues in Rotational Crops*.

**Use-site categories and DACO**

Metabolism/residues in rotational crops studies are conditionally required for use-site categories 10, 13 and 14, and must be submitted to the PMRA under DACO Part 7.4.3.

These studies are not required for use-site categories 1, 5 and 8.

**References**

OECD (2007). Test Guideline No. 503: Metabolism in Livestock, OECD Guidelines for the Testing of Chemicals, Section 5, OECD Publishing, Paris. https://doi.org/10.1787/9789264061873-en

OECD (2007). Test Guideline No. 501: Metabolism in Crops, OECD Guidelines for the Testing of Chemicals, Section 5, OECD Publishing, Paris. https://doi.org/10.1787/9789264061835-en

OECD (2007). Test Guideline No. 502: Metabolism in Rotational Crops, OECD Guidelines for the Testing of Chemicals, Section 5, OECD Publishing, Paris. https://doi.org/10.1787/9789264061859-en

OECD (2018). Guidance Document on Residues in Rotational Crops, Series on Pesticides No. 97 and Series on Testing & Assessment No. 279, OECD Publishing, Paris. https://www.oecd.org/chemicalsafety/guidance-document-on-residues-in-rotational-crops-99457f3f-en.htm

Health Canada (2018). Guidance for developing datasets for conventional pest control product applications: data codes for parts 1, 2, 3, 4, 5, 6, 7 and 10. https://www.canada.ca/en/health-canada/services/consumer-product-safety/reports-publications/pesticides-pest-management/policies-guidelines/guidance-developing-applications-data-codes-parts-1-2-3-4-5-6-7-10.html

# 7.0 Nature of the pesticide residues in processed commodities – High temperature hydrolysis

Although not a data requirement in Canada, the nature of the pesticide residues in processed commodities under hydrolytic conditions is used as supplemental data. If the study has been conducted by the applicant for another jurisdiction, then it is recommended to submit the hydrolysis data to the PMRA.

Studies on the nature of the pesticide residues in processed commodities are conducted to predict the degradation pathway of the active ingredient under hydrolytic conditions, to identify the degradation products, and to determine the relative amount of degradation products.

Three representative hydrolytic conditions should be investigated that simulate typical commercial food processing procedures based on pH, temperature and time (for example, pasteurization; baking, brewing, boiling; steaming/dehydration; and sterilization). The radiolabelled active ingredient is used to elucidate the possible degradation pathways and for quantitation of the extent of degradation. The use of tritium ($^{3}H$) as a radiolabel is not permitted due to the possibility of hydrogen exchange with water.

Samples may be analyzed directly by chromatography or may be extracted with a series of solvents or solvent mixtures with various polarities and other characteristics depending on the nature of the expected residues. Extractable residues are characterized and identified. Ideally samples should be stored at/or below -18°C. The report should include the routes of degradation observed, the degradation pathway, the composition of TRRs, the limit of quantitation for radioactivity determination and chromatographic separation.

**Test guideline**

For further information, refer to OECD Test Guideline No. 507, *Nature of the Pesticide Residues in Processed Commodities*.

**References**

OECD (2007). Test Guideline No. 507: Nature of the Pesticide Residues in Processed Commodities – High Temperature Hydrolysis, OECD Guidelines for the Testing of Chemicals, Section 5, OECD Publishing, Paris. https://doi.org/10.1787/9789264067431-en

# 8.0 Residue definition

Pesticide residues are the combination of the pesticide and the metabolites, degradates, and other transformation products in/on human foods, feedstuffs, and/or drinking water. The number of distinct chemical compounds in the residue definition may vary significantly from pesticide to pesticide. In some cases, only the parent compound may be found in/on treated commodities, while other pesticides may breakdown, partially or completely, into several metabolites. If there is evidence (in other words, scientific studies) to show that the metabolite is a naturally occurring compound, less toxic than the parent compound or not of toxicological concern, it may be possible to exclude this metabolite from the residue definition.

For each pesticide used on food or feed commodities, regulatory authorities need to determine which residues will be used for (i) dietary risk assessment and (ii) setting and enforcing MRLs. The term "definition of residue" or "residue definition" is used to refer to those residues chosen for these two regulatory purposes.

Residue analysis for risk assessment emphasizes analysis of the parent compound and the toxicologically significant metabolites, taking into consideration both exposure and toxicities. Residue analysis for MRL enforcement purposes focuses on those analytes which would indicate a possible misuse of the pesticide and which also can be detected and measured by a broad base of national laboratories, in other words, residues which are easy to measure (ideally by a multi-residue method). These residues must preferably be present at quantifiable levels, and be common to all commodities in which residues are expected. An enforcement method based on one analyte allows greater utility by compliance authorities and minimizes the need to obtain expensive reference compounds. As such, the residue definition for enforcement purposes should be a single compound (also known as a marker compound) to the extent possible.

Residue definition guidance balances these requirements (in other words, risk assessment and enforcement of MRLs) so that the appropriate chemical moieties can be analyzed and used by applicants generating residue data and by regulatory authorities reviewing such data.

**Test Guidance Document**

For further information, refer to the OECD *Guidance Document on the Definition of Residue* (as revised in 2009).

**Reference**

OECD (2009). Guidance Document on the Definition of Residue (as revised in 2009), Series on Pesticides No. 31 and Series on Testing & Assessment No. 63. OECD Environment, Health and Safety Publications.
https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2009)30&doclanguage=en

# 9.0 Residue analytical methods

## 9.1 Data-gathering and enforcement methods

Residue analytical methods that determine all components of the residue definition for a pesticide are required to assess dietary exposure and to establish and enforce MRLs. They are required for crops (human food and feedstuffs), livestock, and processed food/feed products.

Analytical methods used to determine the magnitude of residues in plant or animal commodities, upon which dietary exposure assessments and maximum residue limits are based, are known as data-gathering methods. Analytical methods used to enforce the MRL(s) are known as enforcement methods. Enforcement methods must be validated by an independent laboratory and extraction efficiency (radiovalidation) must be adequately demonstrated.

The analytical method(s) must be described in a stepwise fashion in sufficient detail to enable competent analysts to use the method, even though they are unfamiliar with the procedure. Enforcement residue analytical methods should be practical, rapid, and quantitate all components of the residue definition.

The applicant should state the limit of detection (LOD) and the limit of quantitation (LOQ) for each of the crops, tissues, milk and eggs. The LOD is defined as the lowest concentration of analyte in a sample that can be detected, but not quantitated as an exact value. The LOQ is defined as the lowest concentration of analyte in a sample that can be quantitated with acceptable relative standard deviation. The applicant should describe how the values for LOD and LOQ were calculated, show sample calculations, and cite any appropriate references.

Possible matrix enhancement or suppression effects of sample co-extractives, on the chromatography system or detection system response can occur and should be addressed. When appropriate, the detection system may be calibrated (matrix-matched standards) using standard solutions in a matrix similar to that of the samples to be analyzed. In addition, appropriate clean-up measures should be incorporated to reduce or eliminate interferences.

## 9.2 Method validation

Validation data must be generated and submitted for all components that may be part of the residue definition for all sample matrices. Typical validation parameters for residue analytical methods include recovery, selectivity, calibration, precision (repeatability and reproducibility), LOD and LOQ. Validation parameters are described in the OECD Guidance Document on Pesticide Residue Analytical Methods (OECD, 2007).

Fortification levels should be appropriate to the expected residues in the RAC. When fortification levels are greater than 0.01 mg/kg and lower or equal to 0.1 mg/kg, recoveries should lie between 70% and 120% of the known quantity of the pesticide and the metabolites fortified into the matrix blanks. Sample to sample standard deviation

should not exceed 20%. Refer to Table 1 of the guidance document on analytical methods (OECD, 2007) when concentration levels are below or above the noted fortification levels, as the acceptable range of % mean recoveries and relative standard deviations vary.

Applicants are to report individual recovery values, standard deviations, and coefficients of variation for the parent compound and the metabolites. If 70% recovery is not attainable, the PMRA will accept, on a case-by-case basis, methods having lower recoveries for active ingredients provided the coefficient of variation (CV) is sufficiently low. For example, a method with an average recovery of 65% and a low CV, for example, 5%, may be more acceptable than a method with an average recovery of 95% and a CV of greater than 20%.

The RAC, processed fraction, tissue, milk, eggs, or a macerate thereof, should be fortified, rather than extracts. The fortified macerate should be mixed and allowed to equilibrate for 30 minutes prior to extraction, or less than 30 minutes if the analyte is unstable or volatile. The portion of the crop to be analyzed is specified in Appendix I of this document.

The analytical method must be validated on each crop for which residue data are generated and a MRL is proposed. In the case of crop group MRLs, the method should be validated at the minimum on the representative crops for the group. The report submitted on the method itself should include recovery data (also known as procedural recoveries) on only a representative number of crops. However, in crop field trial reports, additional validation data (also known as concurrent recoveries) should be provided for all crops. For all method validations, at least three samples should be fortified at each level used, to enable a statistical assessment of the method performance. Fortification levels must include the LOQ level for each analyte.

With respect to animal commodities, validation data are required for milk, eggs, and all tissues for which residue data are collected in feeding studies and/or for which MRLs are established. The tissues normally include cattle muscle, fat, liver, kidney, and poultry muscle, fat and liver.

The PMRA accepts the addition of an internal standard to the final extract just prior to injection to serve as a calibration for retention times and/or peak heights/areas and to improve the precision of quantitation. However, the use of an internal standard throughout the entire procedure to correct for recoveries is not acceptable unless data are available on numerous samples of each matrix to show that the analyte and the internal standard behave identically in each step.

For chromatographic methods, the peaks for the analyte and internal standard should elute close to one another but be resolved from each other. As with any other reagent or reference standard used in an enforcement method, the internal standard must be available to enforcement laboratories.

Procedural standards are standards that are generated by subjecting the reference standard to some or all the sample preparation procedures specified in the method. The PMRA will accept methods using procedural standards generated from a derivatization

step under certain conditions. If the standard is unstable or cannot be provided, the applicant must provide data to demonstrate the efficiency and reproducibility of the procedure.

## 9.3 Requirements for enforcement methods

One or more of the methods proposed in the application must be acceptable to enforce the MRLs. The enforcement method should be as simple as possible to decrease the cost of monitoring for pesticide residues. General requirements are described in the OECD Guidance Document on Pesticide Residue Analytical Methods (OECD, 2007).

Although certain gas and liquid chromatographic detection systems possess inherent specificity, methods based on these systems should usually be supplemented by a confirmatory method. In general, confirmation by mass spectrometry is suitable. For methods that do not use mass spectrometry, a validated confirmatory method is required to demonstrate the selectivity of the primary method, to ensure that it detects the correct analyte (analyte identity) and that the analyte signal is quantitatively correct and not affected by any other compound. For methods that use mass spectrometry (GC-MS and LC-MS), confirmation of the identity of the analyte can be conducted simultaneously by monitoring at least two additional fragment ions for GC-MS and LC-MS and one additional ion for GC-MS/MS and HPLC-MS/MS. The specificity may also be enhanced using special extraction/clean-up procedures, derivatization, parallel and/or alternate columns.

Provided that a specific confirmatory method is available, the PMRA will not require that an interference study be conducted to show whether other pesticides registered on the same commodities interfere with determination of the residue definition.

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

**Enforcement method in animal commodities requirement**

Even when a use results in no expectation of quantifiable residues in animal commodities (meat, milk and egg) from feeding of treated feed or direct application, an enforcement method in animal commodities is required in order to establish MRLs at the LOQ of the enforcement method.

**Enforcement method not required in feed commodities**

For crops that are only feed items (for example, forage grasses, pasture), an enforcement method in such plant matrices is not required as MRLs are not established in feed crops in Canada. A validated data-gathering method is required for crops that are only feed items.

## 9.4 Independent laboratory validation of enforcement analytical methods

An independent laboratory validation (ILV) is required to demonstrate the reproducibility of the enforcement analytical method. To be approved for MRL enforcement, an analytical method must be reproducible and suitable for use in federal and provincial laboratories throughout the country.

Results of an ILV for analytical methods are required for the compounds that are included in the residue definition for enforcement and must accompany the following types of applications:

a) The first MRL request for residues of a pesticide in a RAC, processed food or animal commodities.
b) Any new MRL request for residues of a pesticide with previously established MRLs if a new method is proposed for enforcement.
c) Any new MRL request for residues of a pesticide with previously established MRLs if the previously approved enforcement method has been significantly modified to accommodate the new commodity. If the applicant is uncertain whether a method change is significant, the PMRA should be contacted.

An ILV is normally not required for confirmatory methods. However, an ILV may be required for confirmatory methods on a case-by-case basis. One instance when the ILV is likely to be needed is for a confirmatory method where the enforcement method is a common moiety procedure that also detects other registered pesticides.

The laboratory personnel, including the study director chosen to conduct the ILV, must be unfamiliar with the method, both in its development and in its subsequent use in analyzing field samples. Provided that this criterion is met, the laboratory chosen to conduct the ILV may be in the applicant's organization.

The laboratory conducting the ILV may contact the developers or previous users of the method prior to running the first set of samples, but all communications must be logged and reported to the PMRA. If the first or second set is not successful, and the laboratory requires additional contact with the developers or other users of the method, all communications should be recorded. Any subsequent additions or modifications to the original method resulting in improved performance should be incorporated into the enforcement method report that is sent to the PMRA.

The ILV should include fortifications at the LOQ and the MRL levels. If the residue levels are low, the LOQ should be 0.01–0.05 mg/kg. The selection of an appropriate LOQ depends

on the analyte/matrix combination. Recovery data should be generated for the following fortification levels: LOQ (5 samples); 10 × LOQ or MRL, whichever is greater (5 samples); and controls (2 samples). However, six samples (three at each fortification level) and one control sample are the minimum.

ILV data should be submitted for a range of one to four RACs selected from each of the commodity categories listed in Annex I of the guidance document (OECD, 2007). The commodity selected should be representative of the category. In the case of commodities with high protein and high starch content, it is not necessary to perform an ILV for a representative matrix of both categories, but rather include one dry (low moisture) commodity in the validation. The commodity for which the applicant had the most difficulty analyzing should be validated. The rationale for the selection of the commodity should be provided. The following animal commodities should be used for ILV: milk, eggs, meat and/or fat, and kidney and/or liver.

For a successful ILV trial, the results on one set of samples, after conducting no more than three sets, must be similar to those achieved by the applicant. Recovery rates should be 70–120% and interference should be negligible compared to the proposed MRL level. Refer to Table 1 of the guidance document (OECD, 2007) for % mean recoveries and relative standard deviations at corresponding fortification levels. If, after three sets of samples, the ILV has failed to produce adequate results (see below), the applicant must revise the method and run a second ILV, using a different laboratory.

If the ILV is successful, the following should be submitted to the PMRA by the applicant:

a) Name, address, and telephone number of the study director and other contact person for ILV laboratory;
b) Description of the analytical method;
c) Recovery and control values;
d) Representative chromatograms/spectra of the untreated sample, treated sample and untreated sample fortified at LOQ and MRL level for each analyte in each matrix;
e) Description of the instruments used;
f) Description of any problems encountered and a written description of any changes or modifications that were made during the ILV;
g) Any steps considered critical, in other words, steps where little variation is allowable or directions must be precisely followed;
h) The number of person-hours required to complete one set of samples;
i) Any contact between the ILV laboratory and the method developers or others familiar with the method, including the reasons for the contact, any changes in the method that resulted, and the time of this communication with respect to the progress of the ILV trial, for example, after the first set, during the second set; and
j) A statement of adherence to Good Laboratory Practice guidance deemed acceptable by the PMRA.

## 9.5 Extraction efficiency

Extraction efficiency determination is also known as radiovalidation. Conventional recovery experiments, as discussed above, do not necessarily reflect the efficiency with

which bio-incurred residues are extracted from plant and animal matrices. There should be some assurance that aged/bio-incurred residues are completely extracted by the analytical procedure.

Analytical methods must be radiovalidated to determine whether the components of the residue definition are extracted from plant and animal tissues, milk and eggs containing bio-incurred $^{14}$C-residues. Radioisotope labelling from the plant and/or livestock metabolism studies provides the best evidence on completeness of extraction, in other words, extraction efficiency must be validated using samples containing $^{14}$C-bio-incurred residues.

The applicant must demonstrate that the extracted radioactivity accounts for most of the residue definition that was identified in the metabolism study. If an analytical method is to be used on both plant and animal commodities, it must be radiovalidated on a plant matrix, an animal tissue, and either eggs or milk. Matrices for which extraction is expected to be most difficult should be used. In the case of plants, this would normally be a dry sample, for example, straw or fodder, containing $^{14}$C-residues. Applicants should provide a rationale for the samples used in the radiovalidation.

Certain components of the residue definition may be bound with naturally occurring plant constituents, and thus may not be recovered by solvent extraction techniques that are satisfactory for the free components. Such information is evident from metabolism studies. Whenever there are indications of the formation of bound components that may not be recovered by the extraction solvent, modifications should be made in the procedure that will free and recover the released components. One such modification would be the initial hydrolysis of the residues in treated crop. These bound components may also be recovered with polar solvents and hydrolyzed under acidic, basic, or enzymatic conditions to free the components. These should not be confused with those fragmentary components that may be so tightly bound or incorporated into the plant's carbon pool that they are not recoverable by any chemical means. Such components are of interest but are not usually of toxicological concern.

## 9.6 Storage stability of standard solutions and stored extracts

It is important that the stability of a standard working solution be demonstrated to ensure that absorption, adsorption and degradation of the standard in solution were not significant during the period required to analyze samples for residues. The stability of working solutions must be explicitly defined to show the relationship between detector response at a given concentration as a function of time stored in the refrigerator or freezer and as a function of time during the day of analysis when the standard solution(s) is maintained at room temperature during determination of the analyte(s). The applicants are encouraged to follow the regulatory requirements for Good Laboratory Practice.

**Analytes in standard solutions (working)**

In general, working solutions (fortification/calibration) are used over a periods of several days or weeks. The test conditions (for example, appropriate solvent systems, ambient temperature or refrigerator, light/dark) should be selected to reflect usual storage

conditions applied within the conduct of analyses. For testing, the stability of the stored solutions (typically in peak area or peak height) should be compared with freshly prepared fortification and/or calibration solutions. The concentrations should be chosen so that potential degradation can be observed. If no concentration dependency is observed, it is not necessary to investigate all concentrations applied. In order to obtain reliable data, at least three injections of stored and freshly prepared solutions should be compared. A graph should be provided for each analyte working solution showing detector response (area units) as a function of time, covering the period required for a given day and over the period of several days, for example, four to five days, during which time analyses were conducted. These data may be extracted from the standard injections that would be part of the determination data collected for residue studies and method validation. If the solvent used for preparation of the stock or working solution is changed, then new graphs illustrating the stability of each analyte in the new solvent must be provided, as discussed above. Detector response factors, in other words, area units/µg or ng at a given attenuation, should be reported for all new working solutions over the period required for analyses of metabolism, residue, crop rotation and feeding studies.

**Analytes in stored extracts of the final volume**

Ideally, the validation samples are analyzed within 24 hours after initial extraction. Under some circumstances, samples may be stored longer under ambient conditions (for example, in the autosampler or in a refrigerator) if the analyses cannot be completed within one working day. In this case, information on the storage stability of the relevant analytes in extracts and in the final volume should be provided.

**Guidance document**

For additional information, refer to OECD *Guidance Document on Pesticide Residue Analytical Methods*.

**Use-site categories and DACOs**

Residue analytical methods are required under all use-site categories and must be submitted to the PMRA under DACO Parts 7.2.1 (Data-gathering method), 7.2.2 (Enforcement method), 7.2.3A (Independent Laboratory Validation) and 7.2.3B (Extraction Efficiency or Radiovalidation).

**Reference**

OECD (2007). Guidance Document on Pesticide Residue Analytical Methods, Series on Pesticides No. 39 and Series on Testing and Assessment No. 72. OECD Environment, Health and Safety Publications.
https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2007)17&doclanguage=en

# 10.0 Stability of pesticide residues in stored commodities

Storage stability data are required to validate the stability or rate of dissipation of all components that may be part of the residue definition (for risk assessment and enforcement) in or on the RAC of plants, processed commodities, or animal tissues, milk and eggs between the time of harvest or sample collection and the final analysis of the residues.

In most instances, samples collected for determining the magnitude of the residue and the nature of the residue, in other words, metabolism, are stored for a period prior to their analysis. During this storage period, residues of the active ingredient and/or the metabolites may be lost by processes, such as volatilization or enzymatic degradation. Therefore, in order to confirm that the nature and level of residues that were present on samples at the time of their collection are the same at the time of analysis, controlled studies are required to assess the effect that sample storage has on all components that may be part of the residue definition. In other words, applicants must show that pesticide residues are stable during frozen storage of analytical samples, or show the degree to which residues decline during that period of time.

The term storage stability in this document does not address (1) manufacturing concentrate product or end-use product storage stability or (2) the storage of food commodities under typical commercial conditions, for example, during the storage and transport of produce prior to reaching the consumer. Studies addressing the latter are examples of reduction of the residues or anticipated residues studies that are occasionally required to obtain a more realistic estimate of residues in food at the time of consumption. The purpose of the present section is to address storage of analytical samples, in most cases under frozen conditions.

Freezer storage stability data are required in conjunction with magnitude of residue studies (for example, crop field trials, processing studies, livestock residue studies and field accumulation studies) if samples are stored frozen for more than 30 days between collection and final analysis (in other words, not just between collection and extraction of the samples). If samples from residue studies are all analysed within 30 days of their storage under frozen conditions, the applicant can omit conducting a freezer storage stability study provided a justification is given (for example, based on physico-chemical properties). Normally, samples should be frozen within 24 hours of sampling or harvest. If the pesticide is known to be volatile or labile, then freezer storage stability data are required. The determination as to what constitutes volatile or labile will be based on information, such as basic physical properties and the results of metabolism studies.

**Analysis of day-zero samples**

When generating freezer storage stability data, analysis of day-zero samples (in other words, analyzing fortified samples on the day they are fortified before storing them in the freezer) must be conducted. It is not equivalent to analyzing a freshly fortified sample when analyzing stored samples at one of the subsequent storage interval.

If day-zero data are not generated, it will not be possible to use the freezer storage stability data to extrapolate to other crops and other magnitude of the residue studies.

## 10.1 Storage stability studies

There are two types of studies where storage stability data are generated, concurrent studies and stand-alone studies.

Concurrent storage stability data are generated as part of a magnitude of residue study. Placing samples with known residue levels into storage along with the treated commodity samples represents good analytical practice. If the treated samples were subjected to erratic storage conditions due to loss of electrical power, the samples with known residue levels could be used as a direct measure of any effects that temperature fluctuations might have on residues. If the concurrent freezer storage stability study does not include day-zero samples (in other words, samples that are fortified and analyzed the same day as the storage samples are prepared and put into the freezer), the results cannot be used to extrapolate to other crops/studies.

Stand-alone storage stability studies will be acceptable if the storage conditions, especially temperature, duration, and matrix form are the same as those in the corresponding residue study. However, for pesticides with residues that are known or suspected to be unstable or volatile, concurrent studies may be needed. In fact, for such pesticides, it is advisable to run a storage stability study in advance of the residue studies to determine proper storage conditions and maximum storage times before treated samples are placed into storage. The applicant should ensure that all residue samples are analyzed within the time window for which the period of stability of the residues of interest has been demonstrated in the commodity.

Freezer storage stability studies should include sufficient starting material and should have a sufficiently high concentration of residue to allow for any observed decline during storage to be quantified. Samples could either be from crops or animals that have been treated with pesticides in the field, or from the fortification of control commodities, in other words, untreated samples with known amounts of each component that may be part of the residue definition. In all cases, the storage stability samples should be analyzed using the same analytical procedure that was employed in the corresponding residue trial studies. If not, data will be needed to show that the method gives results equivalent to those obtained by the method used in the residue trial studies. The samples used in the storage stability study could also be those obtained from metabolism studies using radiolabelled material. If these are to be used, the residues should be measured using the analytical method that was employed in the residue trial studies or another method validated for quantitating the residue definition. In other words, the storage stability data should not be based on simply counting total radioactivity. Regardless of the method used, freshly fortified control samples of the stored commodities should be analyzed at each of the time points when aged/stored commodities are removed from frozen storage for analysis. These freshly fortified control samples are used for procedural recovery determination (in other words, to verify if the analytical method is adequate) and must represent all the same commodities as the storage stability samples. This will allow for correction of observed residue values for the stored samples if recoveries are

significantly higher or lower than 100% for the freshly fortified samples. At least two sampling intervals (time zero and one other) should be used in a freezer storage stability study; the sampling interval depends on the stability of the residues. Duplicate samples of every commodity at each time point for all components (in other words, analytes) that may be part of the residue definition need to be analyzed. The report should include the residue results and the statistical treatments.

**Additional considerations**

Analytical methods yielding low and variable recoveries should be avoided when conducting storage stability studies as well as the residue trial studies. In those instances where no detectable residues, or low levels of residues close to the analytical method's LOQ are found in field treated commodities, the PMRA advises that fortified control samples be employed in the storage stability studies. Related to this point, it is suggested that the residue level to be used in storage stability studies be 10-fold the method's LOQ with the minimum to be 0.1 parts per million (ppm). This will make it less likely that the stability of the residues cannot be ascertained due to highly variable recoveries. If typical residues observed in the residue trial studies are much higher than the minimum level suggested above, it is preferable, although not required, for the storage stability study to employ comparable residue levels.

In those instances where the residue definition consists of more than one component, in other words, parent compound plus metabolite(s), the storage stability samples may be fortified with the mixture if the analytical method is capable of measuring each component of the residue separately. In those cases where the method converts all residues to a common moiety, fortification with mixtures or using field treated/weathered residues is discouraged. The type of chemical and toxicity involved would determine the acceptability of fortification with a mixture or using field treated samples when a common moiety method is employed. For example, with pesticides where similar chronic toxicity concerns exist over numerous components of the residue, fortification with a mixture followed by use of a common moiety method is probably acceptable. On the other hand, it would not be acceptable to use a common moiety method for cholinesterase inhibitors where significant differences in toxicity may occur as the parent compound oxidizes to assorted metabolites. In other words, in the latter case the method would need to detect each of the metabolites separately.

## 10.2 Plant commodity categories

In the cases of studies involving crop commodities, the principles of extrapolation between commodities within specific commodity categories is recommended. The commodity categories are as follows: (1) commodities with high water content; (2) commodities with high acid content; (3) commodities with high oil content; (4) commodities with high protein content; and (5) commodities with high starch content. It is recognized that some commodities can fit in more than one category (refer to Annex 1 of OECD TG 506).

If residues are shown to be stable in all commodities studied, a study on one commodity from each of the five commodity categories is acceptable. In such cases, residues in all

other commodities would be assumed to be stable for the same duration of time under the same storage conditions (in other words, the longest demonstrated interval common to all of the five categories). If there is no observed decline of residues across the range of the five different commodity categories, then specific freezer storage stability data for processed commodities will not be needed. As such, the demonstrated interval of storage stability applicable to RACs could also be extrapolated to processed fractions.

The crop parts to be examined in these studies are those used for food and feed, in other words, those on which residue data are generated and MRLs established (for example, wheat grain) or the magnitude of feed residues are assessed (for example, wheat forage and wheat straw).

If uses are sought in one of the five commodity categories, then residue freezer storage stability data beyond one representative commodity in that category will be needed (with the exception of the high protein category, which has only one commodity type).

A freezer storage stability study on commodities in the corresponding category must be conducted in accordance with the following requirements:

**High water content category:** If the stability of test substance in **three** diverse commodities in this category is confirmed, further examination with other crops that belong to this category is unnecessary.

**High oil content category:** If the stability of test substance in **two** diverse commodities in this category is confirmed, further examination with other crops that belong to this category is unnecessary.

**High protein content category:** If the stability of test substance in dry legume/pulses is confirmed, further examination with other commodities that belong to this category is unnecessary.

**High starch content category:** If the stability of test substance in **two** diverse commodities in this category is confirmed, further examination with other commodities that belong to this category is unnecessary.

**High acid content category:** If the stability of test substance in **two** diverse commodities in this category is confirmed, further examination with other commodities that belong to this category is unnecessary.

The above guidance is directed toward a pesticide that will be applied to many crops or crop groups within the five commodity categories. However, pesticides may be proposed for use only on a few crops/crop groups. Therefore, the five commodity categories listed above will not always be the most appropriate ones. Since the PMRA cannot provide guidance for all the possible combinations of crops that might be treated, applicants will need to use judgment as to which representative commodities they should use for storage stability studies. Applicants may contact the PMRA if questions arise as to which commodities should be tested for a particular combination of treated crops.

If residues are found to be unstable in any representative commodity, storage stability studies will be required on additional commodities of that commodity category if MRLs are being sought on such crops. Under these circumstances, the concept of commodity category may no longer be applicable.

## 10.3 Storage stability in processed commodities

Freezer storage stability data are required for each of the processed commodities if samples are stored frozen for more than 30 days between collection and final analysis (in other words, between harvest of the RAC and processing, then extraction and until analysis of the samples).

As noted in Section 10.2 above, if there is no observed decline of residues across the range of the five different plant commodity categories, then specific freezer storage stability data for processed foods or feeds will not be needed. However, if instability is shown after a certain length of storage, the applicant should ensure that any commodities (RAC or processed commodity) are analyzed within the demonstrated storage stability time period.

## 10.4 Storage stability in animal matrices

With respect to animal commodities, storage stability data are required to support livestock feeding or dermal treatment studies if samples are stored frozen for more than 30 days between collection and final analysis. The representative commodities to be examined should include muscle from cattle or poultry, liver from cattle or poultry, milk, and eggs. If residues are stable in these matrices, analyses of other tissues, such as fat and kidney, will not be needed.

**Test guideline**

For more information, refer to OECD Test Guidelines No. 506, *Stability of Pesticide Residues in Stored Commodities*.

**Use-site categories and DACO**

Freezer storage stability studies are conditionally required for all use-site categories and must be submitted to the PMRA under DACO Part 7.3.

**Reference**

OECD (2016). Test Guideline No. 506: Stability of Pesticide Residues in Stored Commodities, OECD Publishing, Paris. https://www.oecd-ilibrary.org/environment/test-no-506-stability-of-pesticide-residues-in-stored-commodities_9789264061927-en

# 11.0 Crop field trials, residue decline studies and crop grouping

The purpose of this section is to provide guidance to applicants in generating crop field trials and residue decline studies. This section will also address cases where crop field trial studies generated on the representative commodities of a specific crop group or subgroup may be used to support registration and the establishment of MRLs on the whole crop group or subgroup.

## 11.1 Crop field trials

Crop field trials (also known as supervised field trials) are conducted to determine the magnitude of the pesticide residues in or on RACs, including animal feed items, and should be designed to reflect pesticide use patterns that lead to the highest possible residues. The field trials attempt to account for the variability in results among field trials by selecting more than one test site. This will measure the combined effects of factors such as soil type, weather, and regional cultural practices. Residue data from only one season are considered sufficient provided the crop field trials are located in the major production areas of that particular crop.

The objectives of crop field trials are:

a) to quantify the expected range of residues in crop commodities following treatment according to the proposed or established Good Agricultural Practice (GAP);
b) to determine, when appropriate, the rate of decline of the pesticide residues over time on crop commodities of interest;
c) to determine residue values such as the Supervised Trial Median Residue (STMdR) and the Highest Average Field Trial (HAFT) residue for conducting dietary risk assessments and calculating the dietary burden of livestock; and
d) to derive MRLs for enforcement purposes.

Crop field trials can also provide the relative and absolute amounts of parent compound and metabolites, information which can be useful when determining the compounds that should be included in the residue definition(s).

The test substance is the product or formulation used in the crop field trials. For all applications, the rate should be expressed in terms of amount of product and/or active ingredient per unit area. At the end of each crop field trial, the (stored) samples are analyzed for residue level (expressed in mg active ingredient/kg plant material or ppm).

The formulation tested in crop field trials should be as close as possible to the intended end-use product for the crop or commodity. General information on types of formulations and application parameters such as spray volume, application rate, timing and frequency, retreatment interval, or presence of adjuvant, are given in the OECD Test Guidelines No. 509 (OECD, 2021).

**Spray volumes – Ground versus aerial equipment**

Provided that the pesticide product label specifies that aerial applications are to be made in a minimum of 20 litres of water per hectare, or 95 litres of water per hectare in the case of tree or orchard crops, crop field trials reflecting aerial application will be waived in those cases where adequate data are available from use of ground equipment reflecting the same application rate, number of applications, and preharvest intervals. This data waiver does not apply to aerial applications using diluents other than water, for example, vegetable oils. In addition, the PMRA reserves the right to require aerial data if special circumstances warrant it. However, there are a few instances where the number of field trials is affected by the spray volumes or type of equipment, for aerial versus ground, specified on the label. Therefore, applicants are encouraged to consult with the PMRA.

Residue data must be generated for all food and feed crop parts according to the proposed use directions. Appendix I of this document lists the RACs, processed commodities, and animal feedstuffs that should be included in the residue trials for each crop. The crop parts to be harvested and analyzed as well as the field sample size are listed in Annex 1 of OECD Test Guidelines No. 509 (OECD, 2021).

Validated analytical methodology, appropriate storage stability data, and documentation on sample handling, shipping, and storage intervals and conditions from sampling to extraction and to analysis are required to support all field trials. Sampling and analysis of treated and control samples for each RAC of a crop must be included in all field trials. Commercially important varieties of a crop, as well as seasonal variations, for example, winter wheat versus spring wheat, must be covered by the field trials. Data on different varieties are especially important if there are significant differences in crop size and/or length of growing season.

### 11.1.1 Harvesting crops

**Preharvest interval (PHI)**

The PHI is the minimum period of time, in days (calendar days, not a 24-hour period), that must elapse between the last application of a pesticide and harvesting of plants, and should refer to human food crops or to the edible parts of crops only (refer to pregrazing intervals and feeding restrictions below as applicable to animal feedstuff). While a PHI may be applicable to animal feed items derived from crops, the terms used are slightly different from food items.

For early postemergence application timing, the time of application is well defined by the growth stage (for example, BBCH, a decimal code system). In this case, setting of a PHI is not necessary. The selection of the results from residue trials depends on the use of the pesticide at the correct growth stage and the normal harvest of the product. A PHI on the pesticide label is not necessary for preplant and pre-emergence timings of application, and for seed treatment uses. When the use instructions on a pesticide label do not have a PHI, crops must be harvested at maturity.

**The crop field trials must report the level of residues at the proposed label PHI. For all trials, both the growth stage at the timing of application and the PHI should be reported.**

**Pregrazing interval and feeding restrictions**

For forage/feed crops, pesticide product labels should specify pregrazing intervals (the period between last application of a pesticide and grazing of animals) or feeding restrictions (intervals for cutting feed items such as hay, forage and straw).

Forage is the terminology used specifically for immature crops (green foliage crops). For example, wheat forage is the immature green plant harvested 30 days after an early postemergence application. Wheat hay (harvested after 45 days and left to dry in the field for a few days before being collected) and wheat straw (harvested at maturity after 60 days or more) are not considered forage (although they are animal feed items).

For some crops, the term "harvest" may be unclear due to different practices. The PHI is the number of days that must elapse between the last pesticide application and harvest. In this context, harvest for cereals, oilseeds and hay crops, includes direct-combining and cutting (swathing); it does not include swathing and combining afterwards, or letting the hay dry on the ground and baling afterwards.

While residues may continue to dissipate in the crop following harvest, the PHI is based on cutting, and not on post-cutting activities.

**Seed screenings and aftermath**

For crops grown for seed production only, there is the potential for the "seed screenings" and/or "aftermath" to be fed to livestock. Aftermath is considered the residual material left in the field after seed harvest. Seed screenings are the material that remains after the seeds are cleaned and are comprised primarily of flower pod pieces, chopped/pulverized pieces of stem and other plant parts. Therefore, for crops that are grown for seed production only, residue data are required for forage and hay. Residues in these commodities are expected to cover potential residues that remain in the seed screenings or aftermath, which are considered animal feed items.

### 11.1.2 Sampling

The crop parts to be harvested and analyzed as well as the field sample size are listed in Annex 1 of OECD Test Guidelines No. 509 (OECD, 2021). In each field trial report, the applicant must indicate whether these guidelines have been followed. If the sampling deviates from these guidelines, an explanation must be provided along with details on any deviation. The applicant must also include in the field trial report, the number of agricultural commodity units making a composite sample as well as the weight of the composite sample.

Two independently composited samples (in other words, duplicates) of a treated commodity must be collected at each plot from each trial site. In addition, each site must also include at least one control plot, in other words, untreated plot. In those cases where the two treated composite samples are obtained from the same plot, the samples should be randomly collected in two separate sampling operations in the plots. Splitting

one sample from a plot or conducting two analyses on one sample is not an acceptable alternative to separately collecting and analyzing two samples. In other words, multiple analyses of a single sample or of subsamples constitute the equivalent of only one sample. However, as explained below, if such multiple analyses are conducted, each value must be reported.

For crops that require only two field trials, four treated samples per trial must be collected. The PMRA encourages applicants to conduct a minimum of three field trials to allow the use of the OECD calculator to determine the MRL.

### 11.1.3 Independence of trials

The PMRA uses the OECD MRL calculator as the statistical tool for determining MRLs. The guidance provided in the *OECD MRL Calculator Statistical White Paper* specifies the use of one data point for each trial. If multiple replicate samples are available, the average field trial value is used in the calculation procedure. The procedure further requires that each average field trial value selected is independent. For trials conducted at the same location, they must be independent if the results for each of the trials will be used for the determination of the MRL.

There are two criteria that may be used to identify crop field trials as independent. Any one of these two criteria identifies the crop field trials as independent:

**1) Geographical location and site.** The trials must be physically separated by some distance. Trials in different NAFTA zones are independent of each other. Trials within a zone are independent if they are separated by sufficient physical distance so as to have (potentially) different/modified soil types, climatic conditions, growing conditions, and/or agricultural practices. No precise distance can be specified, but evidence of a difference in growing conditions must be supplied for trials separated by less than 30 km. A separation of 30 km or more will be considered proof of independence.

**2) Dates of planting (annual crops) and pesticide applications.** Replicate plots (at a given site) may be independent if the timing of planting or the initial application of the pesticide is at least 30 days apart. The concept is to have application of the pesticide at the same growth stages of the crop, but in different segments of the growing season. Trials at the same location in different seasons or years are independent (for both permanent and annual crops). Within a season, replicate plots are independent if the growing periods are separated by sufficient time to encompass different climatic or production conditions. The time separation must be relevant to the GAP. For example, time of planting or time of application (within one growing season or year) might not be relevant for a pesticide with a 0–1 day PHI. It is the responsibility of the applicant to explain the relevance of the timing to the independence of the trials.

Additional factors may influence the independence of trials and may be taken into consideration on a case-by-case basis. These may include, but are not limited to, such factors as crop variety, formulation type, application rates and spray concentrations, application equipment, and the addition of adjuvants. Applicants are strongly

encouraged to design the number of independent trials based on the factors of geographical location and timing of application.

Furthermore, each plot must receive independently prepared applications of the pesticide to allow assessment of variability. In other words, the same tank mixture (for example, pesticide treatment, spray solution, preparation, batch) must not be used to treat more than one plot.

## 11.2 Crop field trial requirements

### 11.2.1 Canadian national registration

The crop field trial requirements used by PMRA to support a national registration of a pesticide on a crop were revised and adopted for studies conducted after January 2012.

### 11.2.2 Number and location of field trials

For the number and locations of field trials, refer to PMRA's Regulatory Directive DIR2010-05, *Revisions to the Residue Chemistry Crop Field Trial Requirements*. The number of trials reflects the crop field trial requirements for **one** formulation type being requested for use on each crop.

The total number of crop field trials required for a given crop is determined by the total production area and the dietary share. The specific locations of the field trials are distributed according to the share of total crop area reported in each region. Updated crop production data were determined as part of the 2006 Census of Agriculture (CEAG).

Several new crops were identified in the 2006 CEAG. These include blackberries, borage, chickpeas, corn (popcorn), garlic, linola, peanuts, sweet cherries, tart cherries, walnuts, and wild rice. As these crops have significant production areas and/or are important contributors to the diet, they were added to the revised crop field trial requirements. In addition, crops grown in greenhouses and growing houses (in other words, cucumbers, lettuce, mushrooms, peppers, and tomatoes) were included.

The numbers of trials are intended to cover terrestrial food and feed uses on growing crops. Due to controlled climatic conditions or specific uses, including postharvest dips of fruits and postharvest treatment of grains, two trials and eight treated samples are sufficient for postharvest uses. For greenhouse crops, four trials are required. Other types of application will continue to be handled on a case-by-case basis.

For the purposes of standardizing the number of required field trials, it should be emphasized that in most cases, the number of trials represents the minimum number of trials that is acceptable, with the exception of uses resulting in non-quantifiable residues (refer to Section 11.2.6) and of crop group MRLs (refer to Section 11.4). Fewer trials are needed for a registration amendment (for example, registration of a different formulation with the same use pattern) provided that the existing MRL is shown to be adequate. Additional trials are always welcome because more data points provide greater certainty of expected residue levels and a more robust MRL.

The numbers of trials in DIR2010-05 represent the minimum number of trials that must be performed at the maximum rate per application and per season, the maximum number of applications, the minimum interval between applications, and the minimum preharvest interval. In cases where multiple use patterns are desired and it is not clear which use pattern would result in the highest residue, for example, different PHIs as a function of the application rate, the full number of trials is needed for each use unless side-by-side studies consistently show higher residues from one use pattern.

Commercially important varieties of a crop, as well as seasonal variations, for example, winter wheat versus spring wheat, must be covered by the field trials. Data on different varieties are especially important if there are significant differences in crop size, for example, small-sized tomatoes versus regular-sized tomatoes, and/or length of growing season. In those situations, residue trials must be conducted using the different varieties split among the required number of trials for the crop.

Greenhouse eggplants: If residue data were generated on greenhouse bell and non-bell peppers, these residue data can be extended to cover expected residues in greenhouse eggplants.

**Determination of number of field trials for crops not listed in DIR2010-05**

**Step 1** Assign a base number of field trials to each crop as follows:

| Area | | Base number of field trials |
|---|---|---|
| Hectares | Acres | |
| > 4 046 860 | > 10 000 000 | 16 |
| > 404 690 ≤ 4 046 860 | > 1 000 000 ≤10 000 000 | 12 |
| >121 410 ≤ 404 690 | > 300 000 ≤1 000 000 | 8 |
| > 12 140 ≤ 121 410 | > 30 000 ≤300 000 | 5 |
| > 810 ≤12 140 | > 2000 ≤30 000 | 3 |
| > 81 ≤810 | > 200 ≤2000 | 2 |
| ≤ 81 | ≤ 200 | 1 |

**Step 2** Increase the base number one level (for example, 8 to 12 or 12 to 16) if the area exceeds 121 410 hectares (300 000 acres) and the dietary share is 0.40% or more. For example, wheat, oats, and potatoes.

**Step 3** Decrease the base number one level if the area exceeds 121 410 hectares (300 000 acres) and the dietary share is less than 0.10%. For example, tame hay, flaxseed, dry field peas, lentils, mustard seed, corn for silage, and canary seed.

**Step 4** Increase the base number one level if the area is 121 410 hectares (300 000 acres) or less and the dietary share is 0.02% or more. For example, this is applicable to all fruits and vegetables, except cranberries, Saskatoon berries, green onions and shallots, Brussels

sprouts, radishes, Chinese cabbage and other ethnic leafy vegetables, leeks, hazelnuts, and filberts.

**Step 5** A minimum of 16 field trials is required for crops of more than 121 410 hectares (300 000 acres) and a dietary share of more than 1.00%. For example, wheat, oats, potatoes. Oats was found to exceed the 1.00% diet criterion when using the infant diet, but not when using the diet of the general population.

**Step 6** A minimum of 12 field trials is required for crops of 121 410 hectares (300 000 acres) or less and a dietary share of more than 1.00%. For example, apples and tomatoes.

**Note:** The American methodology includes an additional step where the base number is reduced by one level if 90% of the crop is grown in one region. This step was omitted from the Canadian Guideline because only one crop, soybeans, would be affected.

### 11.2.3 Proportionality concept

Proportionality is defined as the direct relationship between the application rate and the resulting pesticide residues. The proportionality concept is based on the assumption that pesticide residues will increase or decrease linearly with the application rate. For more details, refer to the OECD Guidance Document on Crop Field Trials (Section 3 – Proportionality).

Trials that reflect use patterns other than the proposed GAP are not counted unless the difference in use is within ± 25% of any **one** component of GAP (for example, application rate). The trials that fall outside this criterion, may potentially be scaled according to the proportionality concept if the number of applications and all the other parameters of the use pattern are the same.

The proportionality concept may be used to adjust residue values relative to application rate for field trials conducted within a rate range between 0.3-fold and fourfold the GAP rate. Hence, a use may be supported by up-scaling residue data from trials conducted at rates below the GAP or by down-scaling residue data from trials conducted at rates above the GAP. This is only valid when quantifiable residues occur in the dataset. Where there are no quantifiable residues, in other words, values are less than the limit of quantitation (<LOQ), the residue value is set at the LOQ. It is unacceptable to scale up in this situation.

### 11.2.4 Request for MRLs on imported commodities

DIR2010-05 addresses only national registration of terrestrial uses on crops grown in Canada. Residue chemistry data requirements for MRLs on imported commodities are similar to those for domestically-grown crops, except for the number and location of trials, which must correspond to the requirements in the exporting country.

### 11.2.5 Canadian growing regions

The Canadian growing regions are identified as follows:

- Zone 1: Appalachian
- Zone 1A: Atlantic
- Zone 5: Southern Ontario
- Zone 5A: Northern Shield
- Zone 5B: St. Lawrence Valley
- Zone 7: Dryland Prairie
- Zone 7A: Southern Alberta
- Zone 9: Rocky Mountains
- Zone 11: Dryland Interior
- Zone 12: Pacific
- Zone 14: Northern Prairie

The PMRA currently considers the following Zones equivalent: 5 = 5A = 5B and 1 = 1A. Provided that the total number of required trials within these zones is conducted, the applicant is free to choose the distribution of trials within these regions. The zone equivalency does not apply to Zones 7 and 7A as these zones are considered distinct because irrigation is used in Zone 7A, but not in Zone 7. Residue trial data from other zones where crops are irrigated can be used to fulfill Zone 7A requirements on a case-by-case basis provided an acceptable scientific rationale is submitted by the applicant.

Refer to Appendix II for a description of crop field trial regions and Appendix III for maps defining Canadian major and minor crop field trial regions.

Some of the Canadian growing regions correspond to equivalent American growing regions, for example, Zone 5. Field trials conducted in the United States, in the equivalent growing region, can be used to fulfill Canadian requirements provided the use pattern is the same as the one proposed in Canada. Residue data generated from outside of North America may be accepted on a case-by-case basis.

### 11.2.6 Uses resulting in non-quantifiable residues

An applicant may decide to conduct 25% fewer trials for crops normally requiring ≥ 8 trials, if metabolism data or field trial data on related crops indicate quantifiable residues are not likely to occur for the proposed use scenario. The possible reductions are:

- 8 trials reduced to 5 trials
- 12 trials reduced to 8 trials
- 16 trials reduced to 12 trials
- 20 trials reduced to 16 trials

The 25% reduction in the number of field trials is acceptable if the following four conditions are met:

1. All the trials show residues are at or below the method's LOQ. Note that, if all of these trials do not show residues below the LOQ, then a full set of trials is required.
2. The method has a sufficiently low LOQ, both from an analytical chemistry standpoint and for risk assessment purposes. This means that the LOQ needs to be in the 0.01–0.05 parts per million (ppm) range in most cases.
3. The trials represent all significant regions of production.
4. No other reduction has previously been applied, in other words, 25% for a major crop within a crop group.

**Crop considerations**

- The reduction is not applicable to crops that require ≤ 5 field trials.
- For crops that have more than one RAC, the 25% reduction for residues below the LOQ may be applied to one commodity even if the others have quantifiable residues. For example, if a pesticide is applied to an early stage of corn, it is possible to find residues on silage, but not in the grain. In this case, 8 trials may be acceptable for grain, even though 12 were required. This is not meant to imply that separate trials are to be conducted for different crop parts. In other words, corn grain and silage are to be collected from each trial site. If no residues are found on grain from a minimum of 8 geographically representative sites, the grain collected at other sites do not need to be analyzed.

To take advantage of this option, applicants must submit adequate recovery data and chromatograms establishing the LOQ of the method.

### 11.2.7 Joint Canada/United States field trial requirements

Guidelines for reduced residue field trial requirements to support joint projects between Canada and the United States were developed and adopted in July of 2017. Refer to *Science Policy Note SPN2017-02, Joint Canada/United States Field Trial Requirements* for these guidelines. The PMRA accepts these reduced residue field trials when the applicant submits to both countries.

### 11.2.8 Exchangeability concept

The global zoning concept for field trial residues relates to the degree to which pesticide residues resulting from a given application scenario in one zone (defined as a geographic, climatic, political, or other zone) differ **systematically** from those in another zone under those same pesticide application practices.

As demonstrated in the published article by Nguyen et al. (2019), if there are no systematic statistically significant differences in residue levels attributable to global zones, crop f000ield trials conducted in one zone could be "exchanged" for or combined with those in another zone with no or minimal effect on the level of the MRL. The exchangeability concept allows residue data from supervised trials conducted in multiple zones/countries worldwide to be used to estimate MRLs. By combining field trial data, a

larger dataset is produced, which is expected to result in a more robust MRL when using the OECD MRL calculator.

When some trials conducted in Canadian representative regions do not meet the Canadian requirement for the number of trials per growing region, the PMRA may accept, on a case–by-case basis, field trials conducted outside of North America, based on the concept of exchangeability of pesticide residue values across geographic regions, provided they reflect the proposed GAP or can be scaled based on the principle of proportionality (see Section 11.2.3, *Proportionality Concept*). The number of regions covered by the submitted trials must provide sufficient geographical representation.

## 11.3 Residue decline trials

The primary purpose of the residue decline trials is to determine the behaviour of residues of the active ingredient and relevant metabolites over time in the treated crop. Residues may increase or decrease in the edible portion of crops as a function of time post-treatment.

The decline in pesticide residues on a raw agricultural commodity may be due to one or more of several factors, principally:

1) physical removal, for example, by rain, wind or volatilization;
2) chemical or photolytic degradation or metabolism in/on the plant; and
3) apparent decline due to crop growth dilution.

### Number of residue decline trials for a given crop

Residue decline data are needed for uses where 1) the pesticide is applied when the edible portion of the crop has formed, or 2) it is expected that quantifiable residues may occur on the food or feed commodities at, or close to, the earliest harvest time.

**For agricultural field uses**, residue decline studies are not required for crops requiring ≤ 3 trials if PHI is > 14 days (required if PHI is ≤ 14 days). The number of decline studies needed is **one** for crops requiring 5–12 trials and **two** for crops requiring 16–20 trials. These studies are included in the 5–12 or 16–20 total trials, in other words, not in addition to these numbers of trials.

For greenhouse uses, one of the four trials must be a residue decline trial.

For fumigation uses, residue data at different time points as a function of the dissipation of residues during/after the aeration protocol are recommended to help mitigate risks of concern when identified.

Number of residue decline studies for a given active ingredient

For most pesticides, it is anticipated that residue decline studies will not be necessary for all crops.

For a given pesticide, additional decline studies are not required within a crop group if studies on representative crop(s) indicate that residues do not increase with longer preharvest intervals. This provides some assurance that the MRLs represent the maximum residues that occur from the use of a pesticide.

If a pesticide is to be applied to all types of crops, decline data must be obtained on the following **five representative commodities**: a tree fruit, root crop, leafy vegetable, grain, and fruiting vegetable. Some flexibility in the choice of crops is permitted. For example, a legume vegetable can be substituted for a fruiting vegetable. However, the crop should be chosen from the representative crops listed for crop groups.

### Test guideline

For guidance, applicants can refer to OECD Test Guideline No. 509, *Crop Field Trial*, OECD *Guidance Document on Crop Field Trials*, and Regulatory Directive DIR2010-05, *Revisions to the Residue Chemistry Crop Field Trial Requirements*.

### Use-site categories and DACO

Crop field trials are required for use-site categories 1, 5, 10, 12, 13 and 14, and must be submitted to the PMRA under DACO Part 7.4.1. These trials are not required for use-site category 8.

Residue decline studies are required for use-site category 5 and conditionally required for use-site categories 12, 13 and 14. These studies must be submitted to the PMRA under DACO Part 7.4.2.

## 11.4 Crop groups

To facilitate the establishment of MRLs, the PMRA uses crop groups. Individual crops are allocated to a crop group based on botanical and taxonomic criteria as well as on cultivation practices. Crop groups simplify the establishment of MRLs by using residue data for crops that are representative of the whole group, extended to all crops within the crop group. There are also subgroups within the crop groups. Each subgroup is a smaller and more closely related grouping of the commodities included in the parent crop group, and the representative commodities for each subgroup are also a smaller subset of those for the parent group.

Several miscellaneous commodities are excluded from the crop group concept because their cultural practices and residue chemistry concerns are distinct from other commodities (for example, peanuts, hops, and cacao beans).

The original crop groups, as well as the revised and newly established crop groups that are currently used by the PMRA, can be found on the Residue Chemistry Crop Groups webpage in the Pesticides section of the Canada.ca website.

**References**


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# 12.0 Seed treatment uses and potato seed-piece applications

The PMRA, in collaboration with the USEPA, performed a retrospective analysis of all seed treatment residue data submitted to the USEPA/PMRA in order to determine whether current data requirements were appropriate, or if some streamlining or tiered approach was possible. As a result of this analysis, a decision tree was developed that details the process for determining the residue chemistry data requirements for seed treatment use (see Appendix I of the Science Policy Note SPN2018-01, *Guidance on Streamlined Residue Chemistry Data Requirements for Seed Treatment Uses and Potato Seed-Piece Applications*).

The outlined procedure differs from current PMRA data requirements as per Regulatory Directive DIR2010-05, *Revisions to the Residue Chemistry Crop Field Trial Requirements* or Science Policy Note SPN2017-02, *Joint Canada/United States Field Trial Requirements* as it:

1) Provides direction concerning data requirements in cases where the seed treatment use is being proposed for a crop that has existing foliar uses of the same active ingredient;
2) Allows for a reduction in the current number of residue trials required for the raw agricultural commodities that are exclusively livestock feed items in cases where field residue trials are required to support the seed treatment use; and
3) Allows for a significant reduction in most of the residue chemistry data requirements in cases where the seed treatment application rate is low.

In lieu of traditional field trial studies, a radiotracer uptake study using the radiolabelled active ingredient can be submitted. When radiolabelled data for a crop grown from treated seed at onefold the application rate show no uptake of residues to the aerial portions and root portion of the crop (for human and/or livestock consumption), in other words, TRRs in all plant tissues are less than 5 ppb, no additional residue chemistry studies are required other than a valid enforcement analytical method. A MRL would then be established based on the analytical method's LOQ, if it is sufficiently low from an analytical chemistry standpoint and for risk assessment purposes. If TRRs are greater than 5 ppb, the standard residue chemistry data requirements would apply. However, uses resulting in no quantifiable residues can be eligible for a reduction in the number of field trial data requirements, if certain conditions are met (see Section 11.2, *Crop Field Trial Requirements*).

The above conditions are not applicable to potato seed-piece application. Due to the unique nature of potato seed-piece application, a separate decision tree was developed (see Appendix II of SPN2018-01) to determine the residue chemistry data requirements for this use pattern. There are two possible scenarios for a potato seed-piece application:

1) Application is a new use for the crop, in other words, there are no currently registered potato uses; or
2) In-furrow and/or foliar uses are already registered. Each scenario requires specific residue chemistry data requirements, which are described in SPN2018-01.

**Test guideline**

Applicants can refer to Science Policy Note SPN2018-01, *Guidance on Streamlined Residue Chemistry Data Requirements for Seed Treatment Uses and Potato Seed-Piece Applications*, for additional guidance.

**Use-site categories**

Seed treatments correspond to use-site category 10.

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# 13.0 Residues in rotational crops (Limited field studies)

Residues in rotational crops from limited field studies (also known as field accumulation in rotational crops, which are considered Tier 2 studies) determine the magnitude of pesticide residues which may accumulate in rotational crops via uptake from the soil (for metabolism in rotational crops, refer to Section 6.3, *Metabolism in Rotational Crops*). The study uses a typical end-use product applied to a field plot. Results of these studies are used to determine whether residues occur in rotational crops that are grown under actual field conditions.

These data may be used to establish crop rotation restrictions (in other words, the time from application to the time when rotational crops can be planted; also known as plantback intervals or PBIs), in the dietary risk assessment, and to determine the need for MRLs in rotational crops.

Studies on field rotational crops are conditionally required for uses of pesticides on terrestrial food/feed crops. A rotational crop use is any field-vegetable crop use, or any other use on which it is reasonably foreseeable that any food or feed crop may be produced after harvest of a treated crop.

Rotational crop studies will not be required for uses of pesticides on the following commodities or crop groups: asparagus, berries, cranberry, ginseng, globe artichoke, grape, mushroom, pome fruits, rhubarb, stone fruits, and tree nuts.

If the level of the TRRs in the confined rotational crops study is equal to or exceeds 0.01 parts per million (ppm) at the desired plantback interval or at 12 months, and once the nature of the residue in the rotational crops is understood, then limited field accumulation trials (Tier 2) must be performed (see Section 6.3, *Metabolism in Rotational Crops*).

The limited field trials must be conducted on three representative crops: root and tuber vegetables; leafy vegetables; and small grains, for example, wheat, barley, oats and rye. The rotational crops must be planted after the minimum plantback intervals that could be expected as part of typical agricultural practice: 7–30 days, 60–270 days, and 270–365 days following the last application of the pesticide. The limited field trials must be conducted in two different North American growing regions, totaling six trials. The pesticide can be applied to a primary crop or to bare soil (which is the preferred option) according to the method specified on the pesticide label, at the maximum proposed/approved seasonal label rate among all the labelled crops. All of the plant parts, described as raw agricultural commodities (food/feed RACs) in Appendix 1 of this document, must be analyzed for the components of the residue definition for rotational crops, if different from that of the primary crops. If some crops could be harvested when immature for consumption (for example, such as young leaf spinach and lettuce), then both immature and mature samples should be collected and analysed.

As with confined studies, soybeans may be substituted for the leafy vegetable. The six trials should be conducted on crops that the applicant intends to have as rotational crops on the label.

If there is no uptake of components of the residue definition in one or two of the representative crops in the confined study, the PMRA still requires six field trials. The trials may be distributed at the applicant's discretion among the representative crops showing uptake. In addition, some of the six trials could be conducted using other crops that are typically involved in crop rotation, such as alfalfa and soybeans. All other study requirements noted above must be complied with.

As with the confined accumulation in rotational crops study, soil should be analyzed after treatment, at time of plantback and at harvest to determine components of the residue definition terminal residues.

If no residues above the LOQ are observed in the RACs in the limited field trials, then no MRLs will be proposed and a plantback restriction will be recommended at the shortest interval tested in the study.

If the limited field studies indicate that quantifiable residues will occur, then extended field rotational crop trials are required (which are considered Tier 3 studies). With respect to treatment, these trials must be conducted in the same manner as the limited trials and follow the same data requirement for the number and location of trials as for primary crops. The design of Tier 3 studies should address the conclusions drawn from Tier 1 and 2 studies concerning the most critical PBI to be investigated, especially for complex residue

situations including relevant metabolites. The concept of super crop groups can be used (refer to the OECD Guidance Document on Residues in Rotational Crops for more details).

If MRLs exist on the crops to be rotated, then rotational data on these crops would be required only if residues in rotated crops are expected to exceed established MRLs.

Therefore, if, in Tier 1 or 2 studies, residues in rotational crops were <0.01 mg/kg at plantback intervals ≥30 days and at appropriate application rates (in other words, after scaling, if necessary), the plantback interval will be established at the shortest interval tested and no MRLs are needed. As a result, Tier 3 (extended field) studies are unnecessary. If in Tier 2 studies, residues in rotational crops reach significant levels (≥0.01 mg/kg), a Tier 3 assessment is necessary to decide on appropriate risk mitigation measures and/or to set MRLs. Studies should be conducted with application to bare soil. Refer to Decision Trees (Figures 1 and 2) on the tiered approach for setting up a rotational crop testing programme in the OECD Guidance Document on Residues in Rotational Crops (OECD, 2018).

**Test guideline and guidance document**

Applicants can refer to OECD Test Guideline No. 504, *Residues in Rotational Crops (Limited Field Studies)*, and OECD *Guidance Document on Residues in Rotational Crops*, for additional guidance.

**Use-site categories and DACO**

Residues in rotational crops (limited field studies) are conditionally required for use-site categories 10, 13 and 14, and must be submitted to the PMRA under DACO Part 7.4.4.

These studies are not required for use-site categories 1, 5 and 8.

**References**

OECD (2007). Test Guideline No. 504: Residues in Rotational Crops (Limited Field Studies), OECD Guidelines for the Testing of Chemicals, Section 5, OECD Publishing, Paris. https://doi.org/10.1787/9789264013384-en

OECD (2018). Guidance Document on Residues in Rotational Crops, Series on Pesticides No. 97 & Series on Testing & Assessment No. 279, OECD Environment, Health and Safety Publications, OECD Publishing, Paris. https://www.oecd.org/chemicalsafety/guidance-document-on-residues-in-rotational-crops-99457f3f-en.htm

# 14.0 Magnitude of the pesticide residues in processed commodities

Studies on the magnitude of the pesticide residues in processed commodities are conducted to provide data on the transfer of residues to different processed commodities from the RAC. The information about reduction and concentration of residues and the estimation of processing factors (the ratio of residue levels in processed commodities to those in the RAC) is used to determine if separate MRLs for processed commodities are required when anticipated residues in the processed commodity exceed the MRL proposed for the RAC; to refine dietary exposure to processed products; and to allow a more realistic calculation of the dietary burden of livestock when processed commodities are used as animal feedstuffs.

Processing studies should measure the active ingredient and metabolites which may be included in the residue definition, as well as relevant major degradation products identified in the "Nature of the Residue in Processed Commodities – High Temperature Hydrolysis" study.

Processing studies are required for all RACs for which processed commodities are listed in Appendix I of this document. At the minimum, a processing study should include all edible processed commodities listed in Appendix I for a particular crop. In some cases, the requirement for a processing study may be waived based on field trial data where residues in the RAC are <LOQ following treatment at exaggerated rates (fivefold the GAP rate).

Processing studies should simulate commercial practices as closely as possible. RAC samples used in processing studies should contain field-treated (bio-incurred), quantifiable residues, so that processing factors can be determined for the various processed commodities. This may require field treatment at exaggerated application rates to obtain quantifiable residue levels. Processing studies utilizing fortified samples are not acceptable, unless it can be demonstrated that the RAC residues consist entirely of surface residues.

At least two independent trials (with RAC samples from two separate field sites) are required as per the OECD TG 508 for each processing procedure to be conducted as long as the commercial procedures are not significantly different for a given commodity (in other words, solvent extraction and cold press for oil production). At least two replicate samples of the RAC should be analyzed. Although OECD TG 508 requires two trials, the PMRA will accept one trial for each crop having processed commodities, as listed in Appendix I of this document. The PMRA however encourages applicants to conduct a minimum of two trials.

When two processing studies are available for a given crop, the mean processing factor is calculated and used to estimate anticipated residues in processed commodities, whereas when three processing studies or more have been conducted, the median processing factor is used in the estimation of residues. Separate processing factors must be determined for each potential component of the residue definition for both enforcement and risk assessment purposes.

The components of the residue definition should be measured in the RAC at the time processing is initiated and in all processed commodities of the crops listed in Appendix I of this document. Refer to Table 1 of OECD TG 508 for a list of processing procedure types and extrapolations using typical RACs.

Unless the processed commodities are stored frozen and analyzed within 30 days of their production, data demonstrating the stability of residues in representative processed commodities during storage are required, as described in Section 10, *Stability of Pesticide Residues in Stored Commodities*.

If the processing of the RAC may result in alteration of the residue, then a radiolabelled processing study to determine the nature of the residue in food, as it is consumed, may be needed. If significant alteration of the residue occurs, and the additional residue components are of toxicological concern, then the MRL should include the additional residue component(s). For more details, refer to Section 7, *Nature of the Pesticide Residues in Processed Commodities – High Temperature Hydrolysis*.

**Test guideline and guidance document**

Applicants can refer to OECD Test Guideline No. 508, *Magnitude of the Pesticide Residues in Processed Commodities*, and OECD *Guidance Document on Magnitude of Pesticide Residues in Processed Commodities*, for additional guidance.

**Use-site categories and DACO**

Magnitude of the pesticide residues in processed commodities are conditionally required for use-site categories 10, 12, 13 and 14, and must be submitted to the PMRA under DACO Part 7.4.5.

These studies are not required for use-site categories 1, 5 and 8.

**References**

OECD (2008). Test Guideline No. 508: Magnitude of the Pesticide Residues in Processed Commodities, OECD Guidelines for the Testing of Chemicals, Section 5, OECD Publishing, Paris. https://doi.org/10.1787/9789264067622-en

OECD (2008). Guidance Document on Magnitude of Pesticide Residues in Processed Commodities, Series on Testing & Assessment No. 96, OECD Environment, Health and Safety Publications, OECD Publishing, Paris. https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2008)23&doclanguage=en

# 15.0 Residues in livestock

Livestock are domesticated animals raised in an agricultural setting to produce commodities such as food for human consumption, and include but are not limited to cattle, pigs, poultry, horses, goats and sheep.

The primary purpose of the residues in livestock studies is to provide the data for establishing MRLs in edible animal commodities and for conducting dietary risk assessments. The residues in livestock studies quantify the transfer of residues into meat, milk, eggs, fat, and edible meat byproducts following indirect or direct animal exposure to a pesticide. The scenarios for which such studies apply include application of a pesticide to RACs that may be fed to livestock; pesticides that may be directly applied to livestock; and pesticides that are used in livestock premises. Human food of animal origin also includes fish and honey (refer to Sections 16 and 17 of this document for more details).

Data from these studies are used to determine which components of the residue definition are present and the magnitude of residues that could result in ruminant meat (muscle), meat byproducts (liver, kidney), fat and milk; and poultry meat (muscle), meat byproducts (liver), fat and eggs.

Separate feeding studies must be conducted for ruminant (lactating dairy cows) and poultry (egg-laying hens). A study for residues in livestock will normally comprise 3 different dose levels, onefold, threefold and tenfold of the anticipated dietary burden. Three animals per dose group (and one for the control) should be used for ruminants. For hens, 9–10 animals per dose group (and 3 to 4 animals for control per study) should be used. The study report should include daily feed consumption, bodyweight measurement, milk or egg production (after and before dosing), detailed observations and tissues analyses.

In the case of pesticides that are directly applied to livestock, residue studies using the prescribed method of application to the livestock species to be tested (dip, spray, pour-on, ear tag, etc.), dosages and withdrawal times, consistent with those of the supported use directions, are required to quantify the magnitude of the residues in edible livestock commodities, as listed above.

When the use of a pesticide in premises such as livestock housings is such that label restrictions cannot preclude the possibility of measurable residues in meat, milk or eggs, residue studies should be conducted reflecting the conditions of maximum exposure.

## 15.1 Dietary burden

The dietary burden is the estimation of total exposure of livestock to a pesticide through feed items expressed in ppm (mg pesticide per kg feed). The Maximum Reasonably Balanced Diet (MRBD), which comprises sources of carbohydrate concentrate [CC], roughage [R] and protein concentrate [PC] will allow livestock to have steady weight gain, high milk volume and consistently high egg production.

The dietary burden calculated for MRL purposes in animal commodities assumes that livestock are exposed to 100% treated crops. As the livestock grow, their nutritional requirements change, and their diet is adjusted to allow adequate growth and maintenance.

The Dietary Burden Calculator is an Excel-based spreadsheet developed by the PMRA to make the livestock dietary burden calculations easier, consistent, and more efficient. The calculator includes the feedstuffs presented in the OECD Guidance Document on Overview of Residue Chemistry Studies (as revised in 2009). Reference livestock include dairy cattle, beef cattle, poultry, and swine. Calculated dietary burdens for beef and dairy cattle can be extrapolated to goats, horses and sheep.

## 15.2 Anticipated residues in animal commodities

Anticipated residues in animal matrices are pesticide residue levels expected in each of the animal commodities when the animal is fed a diet at the dietary burden level. Refer to Appendix I of this document for a list of feed items.

The anticipated residues in animal matrices are used to conduct/refine the dietary risk assessment.

The PMRA has adopted an empirical model based on the Langmuir Isotherm to fit dose-response curve used in dietary burden calculations.

Langmuir Equation:

$$R = R_{max} \ \frac{D}{D+\alpha}$$

Where,

| | |
|---|---|
| R | Residue (mg/kg) |
| $R_{max}$ | max residue (mg/kg) |
| $\alpha$ | half saturation residue (mg/kg) |
| D | Dose (mg/kg) |

The above model is a simplified form of the Hill-Langmuir model. The model is based on the concept that there is a finite number of sites the chemical can bind to so that achieving complete saturation would require a very high dose. A kinetic adaptation of the Langmuir equation results in the Michaelis-Menten model to describe the equivalent saturation effect on enzyme-mediated reaction rates.

Residue values from the livestock feeding studies or animal metabolism studies are used to estimate the anticipated residues in meat, meat byproducts, milk and eggs, while taking into account the residue definition in livestock matrices for MRL/enforcement and/or dietary risk assessment purposes. When quantifiable residues are observed in livestock matrices, anticipated residues are calculated using the results from the livestock feeding study by incorporating them into the Langmuir model. The model is fitted to the small number of data points from dose-response studies on livestock or poultry and is used

to predict residues arising from a given dietary burden. The curve fitting uses the Generalized Reduced Gradient non-linear algorithm in the Microsoft Excel Solver to estimate parameters $R_{max}$ and $\alpha$. The fit to data is as good as the original linear method and consistently better when data shows saturation at high doses.

**Test guideline and guidance document**

Applicants can refer to OECD Test Guideline No. 505, *Residues in Livestock*, and OECD *Guidance Document on Residues in Livestock*, for additional guidance.

**Use-site categories and DACO**

Studies for residues in livestock from feeding of treated crops are required for use-site category 13 and conditionally required for use-site categories 10, 12 and 14. These studies must be submitted to the PMRA under DACO Part 7.5. These studies are not required for use-site category 5 as there are no feed items associated with greenhouse uses.

Studies for residues in livestock from external application are required for use-site categories 1 and 8, and must be submitted to the PMRA under DACO Part 7.6.

**References**

OECD (2007). Test Guideline No. 505: Residues in Livestock, OECD Guidelines for the Testing of Chemicals, Section 5, OECD Publishing, Paris. https://doi.org/10.1787/9789264061903-en

OECD (2009). Guidance Document on Overview of Residue Chemistry Studies (as revised in 2009), Series on Pesticides, No. 32; Series on Testing and Assessment, No. 64. https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2009)31&doclanguage=en

OECD (2013). Guidance Document on Residues in Livestock, Series on Pesticides No. 73, OECD Environment, Health and Safety Publications, OECD Publishing, Paris. https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2013)8&doclanguage=en

# 16.0 Residues in water, fish and irrigated crops

These studies are used by the PMRA to determine the levels of pesticide residues in water, fish, and irrigated crops when pesticides are applied directly to water (excluding chemigation). The data are used in dietary risk assessments and, in the case of fish and irrigated crops, to establish MRLs.

Chemigation labeling and residue data requirements are covered under PMRA Regulatory Directive DIR93-13, *Chemigation*.

Pesticides that are used in or near aquatic sites, including rivers, ponds, lakes, and irrigation canals may result in pesticide residues in water, fish/shellfish, irrigated crops, and animal commodities. Pesticide residues may also be present in water

when fields are flooded and drained as a part of normal agricultural practice, whether before, during, or after treatment with pesticides. When residues are present in water, it may result in the indirect transfer of residues in fish/shellfish, and in meat, milk, and eggs when animals drink from the treated water source. The treated water, from irrigation canals or any other treated water source, can be used to irrigate crops and result in transfer of pesticide residues to these crops. For these irrigated crops, adequate residue chemistry data are required to demonstrate both the nature of the residue and the level of residues in the irrigation water and the irrigated crops resulting from the maximum proposed use pattern.

The design of field studies to demonstrate the fate of the pesticide in the aquatic environment must be directly related to the use pattern and restrictions imposed on the use. Because of the nature of aquatic uses, emphasis must be placed on the practical use restrictions that will be followed by the applicator. In the case of fields treated either before or after flooding, the timing, volume, and release of the flood water as dictated by normal agricultural practice must be considered in the field study design. As another example, use in impounded bodies that are completely under the control of the user may be subject to practical label restrictions that would preclude livestock watering, fishing, or use for drinking or irrigation for a specified time period after treatment. On the other hand, such restrictions would not be practical for the use of a pesticide in a river system. In this type of use, restrictions against treatment within a given distance of irrigation or domestic water intakes may be required.

In general, separate protocols will be required for still waters (in other words, lakes and ponds), flowing waters, irrigation conveyance systems, fields that are flooded and drained, and tidal estuaries. The fate of the compound must be demonstrated with respect to rate of dispersion downstream, degradation, volatilization, or sorption by plants or hydrosoil. Degradation products in water must be identified and quantified.

## 16.1 Residues in water

Residue data are required for any water, as described above, in the various aquatic systems that may either be directly or inadvertently impacted by a pesticide use, in other words, pond, field, drainage canal, river, or estuary. The data collected must show the highest residue level likely to occur in water. If a monitoring scheme is used, it should include samples taken prior to treatment with pesticides and then over time to show the decline of the pesticide residues.

Unless covered off by environmental fate studies, residue data must be provided for treated water at or near the point of application as a function of time posttreatment, until a decline (three data points or decline curve) in the residue concentrations in water is observed.

If residues are likely to occur in treated water and the treated water could be ingested by livestock, then the magnitude of the residues in water will be used in the calculation of the dietary burden of livestock. To determine the transfer of residues to meat, milk and

eggs, feeding studies carried out as described in Section 15, *Residues in Livestock,* will be used.

## 16.2 Residues in fish/shellfish

Pesticide residues can be transferred to fish from aquaculture (direct treatment of fish for ectoparasites) or from aquatic use of a pesticide where fish/shellfish are exposed to treated water from the different scenarios described above. The expected maximum concentration of pesticide residues in fish and the treated water must be quantified.

A fish metabolism study on a predator, such as bass, or bottom feeder, such as catfish, is required when fish may be exposed directly or indirectly to the pesticide or its degradation products. If no radioactivity is detected in fish in a static metabolism study, then additional fish residue studies are not required. However, shellfish residue studies will still be required following exposure to the treated water.

The fish and shellfish residue studies may be of various types depending on the aquatic system involved. Controlled exposure for appropriate time intervals may be carried out under static or dynamic conditions in aquaria, or the specimens may be exposed in natural sites if the treated area can be isolated, for example, by cages. Field studies under natural conditions are preferred. The fish commodities to be sampled and analysed are the skin and the muscle. The proposal for MRLs in fish should be expressed on the basis of the edible portion. For the scenario of treated water, fish residue data are required for both bottom feeders, such as catfish, and predators, such as bass. For shellfish, data are required for both molluscs (for example, clams and oysters, and crustaceans, such as shrimp and crabs). If use in estuarine areas is planned, data may be required in processed fish products.

## 16.3 Residues in irrigated crops

First and foremost, the expected maximum concentration of pesticide residues, including degradation products, in the treated irrigation water must be quantified.

If it is determined that quantifiable residues are expected in the treated irrigation water, studies to determine potential residues in crops that have been irrigated with the treated water are required. These residue trials may utilize the crop grouping scheme with residue data required for the representative crops of each crop group. Applicants are encouraged to consult with the PMRA on the number and location of trials.

**Test guideline**

New OECD guideline on aquaculture is in the future workplan and once completed, will replace part of this section.

**Use-site categories and DACO**

A fish/shellfish metabolism study is required for use-site category 1 and must be submitted to the PMRA under DACO Part 6.2., and fish/shellfish residue data must be submitted under DACO Part 7.4.1.

**References**

FDA (1994). Pesticide Analytical Manual, Vols. I and II, Food and Drug Administration, Washington, D.C. Available from the National Technical Information Service, Springfield, VA 22161.

USEPA (1996). Residue Chemistry Test Guidelines: OPPTS 860.1400 Water, Fish, and Irrigated Crops [EPA 712–C–96–178]. https://www.regulations.gov/document?D=EPA-HQ-OPPT-2009-0155-0010

PMRA (2013). Regulatory Directive DIR93-13, Chemigation. https://www.canada.ca/content/dam/hc-sc/migration/hc-sc/cps-spc/alt_formats/pacrb-dgapcr/pdf/pubs/pest/pol-guide/dir/dir9313-eng.pdf

# 17.0 Residues in honey

Pesticide residues can be transferred to honey from direct treatment of beehives (to control mites) or from treatment of melliferous crops.

**Direct treatment of honeybee hives**

Applicants are encouraged to consult with the PMRA on the residue chemistry data requirements.

**Treatment of melliferous crops**

New OECD guidelines are being developed for residues in honey from treatment of melliferous crops and will be adopted once finalized.

Applicants are encouraged to refer to the OECD Publications on pesticide residues webpage at: https://www.oecd.org/env/ehs/pesticides-biocides/publicationsonpesticideresidues.htm for any guidance on residues in honey that may have been issued after the publication of this document.

# 18.0 Food handling establishments

Studies must be conducted to determine residues resulting from pesticide treatment of food handling establishments.

A food handling establishment is an area or place where food is held, processed, prepared and/or served. Food areas of food handling establishments include areas for receiving; serving; storing of dry, cold, frozen or raw foods; packaging, such as canning, bottling, wrapping and boxing; preparing, such as cleaning, slicing, cooking and

grinding; edible waste storage; and enclosed processing systems, such as mills and dairies, or those systems used to produce edible oils and syrups. Non-food areas of food handling establishments include garbage rooms, lavatories, floor drains to sewers, entries and vestibules, offices, locker rooms, machine rooms, boiler rooms, garages, mop closets, and storage areas for canned, bottled or packaged products.

**Table 1 lists the categories of commercial food handling establishments.**

| Table 1. Categories and representative types of food handling establishments | |
|---|---|
| **Category** | **Representative types** |
| Food services[1] | Restaurants, cafeterias, taverns, delicatessens, mess halls, school and institutional dining areas, hospitals, mobile canteens, vending machines, grocery stores and markets. |
| Manufacturing establishments[2] | Candy plants, ice cream plants, pasta plants, food mix plants, breakfast cereal plants, bakeries, breweries, wineries, soft drinks, bottling plants, pizza plants. |
| Processing establishments[3] | Meats, poultry, and seafood slaughtering and/or packing plants, spice plants, edible fats and oils plants, fruit and vegetable canneries, pickle factories, beverage, e.g., coffee or tea, plants, frozen fresh food plants, grain mills, dairies. |

Data obtained from studies conducted in two different types of establishments in each category is usually adequate to extend to the remaining establishments of that category. The representative types being tested should be carefully chosen to accurately represent that category. More than two types of establishments may need testing depending on the case scenario.

Existing sanitation programs, practices, and the type of building construction, for example, wood, cement block, at a plant site should be considered.

Usage will normally involve the application of the pesticide onto surfaces or into the air of structures as either a surface or space application.

1) Surface application – A directed application to a surface (floor, wall, foundation, ceiling, etc.). This includes but is not limited to broadcast, perimeter, spot, crack and crevice and void applications.
2) Space application – An application of a pesticide as a suspension of fine droplets in air within an indoor space. This definition does not include fumigants.

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[1] Any food handling establishment whose principal business involves the sale of food directly to the consuming public. The manufacture and/or processing of food by such an establishment is only incidental to achieving its principal business objective.

[2] Any food handling establishment whose principal business involves the production and/or packaging of human-made foods that are normally intended for sale through or by food service establishments. Such foods are generally composed of two or more ingredients that have been altered in such a manner as to change their basic identity.

[3] Any food handling establishment whose principal business involves the upgrading and/or preservation of raw agricultural commodities in such a manner as to maintain their essential identity. Such establishments may sell their product directly to the consuming public and/or food service or handling establishments.

This will include food areas of the establishment that is used as the test site.

Acceptable results from a study of the most rigorous type of treatment (space > general > spot > crack and crevice) should cover the less rigorous treatments and additional residue studies would not be required. The study will also allow registration of the pesticide for use by the less rigorous method(s). In many cases, one thorough study representing a worst-case scenario for residues will suffice to cover use in all types of establishments.

Applicants are advised to submit a protocol before initiating a residue study that is intended to support use in food handling establishments. The treatment of establishments for the purposes of this study should be performed in accordance with the proposed labelling.

The study should be designed to reflect all possibilities of contamination. The physical and chemical properties of the pesticide, the proximity of foods and the protective barriers should be considered.

Many practical sources of contamination may be eliminated or diminished through restrictions, variations in the mode of application, the type of establishment treated or the nature of the product or formulation. Data should be submitted to establish the relative importance of these factors on the levels of residue that may be expected to result from pesticide application. Experiments should be conducted by the analyses of representative foods that are subjected to exposure by any of the above routes that are potential avenues of contamination.

The selection of samples for analyses in specialized uses, for example, flour mills, would be apparent. For generalized exposures, for example, grocery stores, the selection of samples analyzed should include a range of foods, such as an oily food (for example, butter); baked cereal products (for example, bread); beverages (for example, milk); raw and processed meats; and fresh fruits and vegetables (for example, lettuce).

To demonstrate the residues resulting from a wide range of conditions and to gauge the potential for misuse, the study should include exaggerated exposure. For example:

a) spraying at twice the rate,
b) exposure of foods for longer periods than expected normally, or
c) exposure of some foods when there is a restriction to cover foods when treating.

**Test guideline**

Applicants can refer to USEPA Residue Chemistry Test Guidelines OPPTS 860.1460, *Food Handling*, for additional guidance.

**Use-site categories and DACO**

Studies depicting residues resulting from pesticide treatment of structures are required for food processing and storage (use-site category 12) and food handling establishments (use-site category 20) and must be submitted to the PMRA under DACO Parts 7.4.1 and 7.4.2.

**References**

USEPA (1996). Residue Chemistry Test Guidelines: OPPTS 860.1460 Food Handling [EPA 712–C–96–181]. https://www.regulations.gov/document?D=EPA-HQ-OPPT-2009-0155-0011

PMRA (2020). Guidance Document, Structural Pest Control Products: Label Updates. https://www.canada.ca/en/health-canada/services/consumer-product-safety/reports-publications/pesticides-pest-management/policies-guidelines/structural-pest-control-products-label-updates.html

# 19.0 Maximum residue limits (MRLs)

The use of pesticides on crops in accordance with the label conditions may result in pesticide residues on human foods and livestock feeds (for example, a use on wheat may result in pesticide residues in wheat forage, hay, grain and straw). In turn, if livestock eat treated feed items, there is the potential for the transfer of pesticide residues into meat, milk, and eggs. Also if livestock receive a direct pesticide treatment (for example, dermal application or ear tags) or are exposed indirectly (for example, treatment of livestock housing), there is the potential for the transfer of pesticide residues into meat, milk, and eggs. Human exposure can result from food consumption of pesticide residues that remain in/on food commodities from plant and animal origins.

The PMRA establishes science-based MRLs for pesticide residues. The MRL represents the maximum amount of pesticide residues that are expected to remain in or on commodities (for example, crop or animal matrices) when a pesticide is used according to label directions. The MRL is expressed in ppm (parts per million) which corresponds to milligrams of pesticide residues per kilogram of food commodities. MRLs are based on residue trials that reflect the maximum residues that may occur under worst-case conditions, in other words, maximum rate per season and minimum preharvest interval/preslaughter interval, as a result of the proposed use of the pesticide.

MRLs are established on specific pesticide-food commodity combinations. The different types of food commodities are:

a) Crops (referred to as raw agricultural commodity or RAC) such as apples, tomatoes, wheat, and soybeans.
b) Processed commodities such as tomato paste, raisins, and canola oil.
c) Animal commodities such as meat, milk, and eggs.

The pesticide active ingredient and any significant metabolites are together called the residue definition (see Section 8, *Residue Definition*, for more details). Separate residue definitions may be established for enforcement and for risk assessment purposes. The residue definition for enforcement must be simple and suitable for practical routine monitoring and enforcement of the MRL at a reasonable cost. The residue definition for enforcement (and MRL setting) often includes only a marker compound, such as the parent compound. When the analytical method is based on the measurement of a common chemical moiety, the residue definition will include the parent compound and

any metabolites converted to the common chemical moiety, expressed as parent equivalents.

**Pesticide:** If there is **no** MRL established for a specific pesticide-crop combination:

1) The pesticide may not be registered for use on that crop. Health Canada's Pesticide Label Search tool can be used to determine if a pesticide is registered for use in Canada; or
2) If the pesticide is registered for use on a specific crop and no MRL has been established, the pesticide residues resulting from the agricultural use on the specific crop are regulated under subsection B.15.002(1) of the Food and Drug Regulations, which requires that residues not exceed 0.1 ppm, commonly known as the General (or Default) Maximum Residue Limit (GMRL). For details, see the excerpt from the Food and Drug Regulations below.

> "Division 15 Adulteration of Food
>
> B.15.002. (1) Subject to subsection (2), a food is adulterated if
> (*a*) a pest control product as defined in subsection 2(1) of the *Pest Control Products Act* or its components or derivatives, for which no maximum residue limit has been specified under sections 9 or 10 of that Act for that food, are present in or on the food, singly or in any combination, in an amount exceeding 0.1 part per million"

**Adjuvants:** Adjuvants are present as formulants in end-use products or can be sold separately and tank mixed with end-use products before application. For more details, refer to Section 5. Chemical Identity. Adjuvants which are sold separately, of the "activator or spray modifier" type, and intended to directly improve efficacy or to enhance biological performance of the end-use product by modifying or enhancing physical or chemical characteristics are subject to registration provisions of the Pest Control Product Regulations (PCPR).

MRLs are not required for adjuvants as they are exempt from the adulteration provision of the *Food and Drugs Act* (FDA), as outlined below.

> "B.15.002 (2)
>
> (2) A food is exempt from paragraph 4(1)(d) of the Act if the following agricultural chemicals, or their components or derivatives, are the only agricultural chemicals, or components or derivatives of agricultural chemicals, that are present in or on the food, singly or in any combination:
>
> (a) a fertilizer;
> (b) an adjuvant or a carrier of an agricultural chemical;
> (c) an inorganic bromide salt;
> (d) silicon dioxide;
> (e) sulphur;
> (f) viable spores of *Bacillus thuringiensis Berliner*; or
> (g) Kaolin."

As MRLs are established under the *Pest Control Products Act* for the purposes of the adulteration provision of the *Food and Drugs Act*, Health Canada does not have the legislative authority to set pesticide MRLs in/on feed items. However, pesticide residues in feed are regulated indirectly by setting MRLs in the food commodities of animal origin (fat, meat, meat byproducts, milk, and eggs) resulting from the transfer of pesticide residues in the animal from eating the treated feed items.

**MRLs on imported commodities:** Applications requesting MRLs to cover residues in any imported foods should contain the same type of data as those required for a Canadian registration. The registered end-use product label from the exporting country and field residue data generated according to the foreign label directions for use are required. A MRL on an imported commodity is required to permit importation into Canada of commodities potentially containing pesticide residues of an active ingredient which is not registered for that use or not registered in Canada.

MRLs established in Canada do not distinguish between residues on a domestically-produced commodity or an imported commodity.

**MRLs on crop groups:** Crop groups simplify the establishment of MRLs by using residue data for crops that are representative of the whole group to extend to all crops within the crop group **(refer to Section 11.4 for** more details**).**

**OECD Maximum residue limit calculator:** With the goal of harmonizing the calculation of MRLs, the OECD has developed a MRL Calculator. It is comprised of a simple Excel spreadsheet which does not require extensive statistical knowledge from the user. There is one spreadsheet for single data sets and one spreadsheet for multiple data sets. A user guide and a statistical white paper are available to help with the use of the calculator. The OECD MRL Calculator Spreadsheets Version 2 were made publicly available on 23 April 2020.

**References**

PMRA (1993). Regulatory Directive DIR93-15, Registration Requirements for Adjuvant Products. https://www.canada.ca/content/dam/hc-sc/migration/hc-sc/cps-spc/alt_formats/pacrb-dgapcr/pdf/pubs/pest/pol-guide/dir/dir9315-eng.pdf

OECD (2011). OECD MRL Calculator: User Guide, Series on Testing and Assessment No. 56. https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2011)2&doclanguage=en

OECD (2011). OECD MRL Calculator: Statistical White Paper, Series on Testing and Assessment No. 57. https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=env/jm/mono(2011)3&doclanguage=en

OECD (2020). Maximum Residue Limit Calculator Spreadsheets Version 2. https://www.oecd.org/env/ehs/pesticides-biocides/oecdmaximumresiduelimitcalculator.htm

# Appendix I

**Raw agricultural and processed commodities and livestock feeds derived from field crops**

A blank space or unnamed fraction in the processed commodity, or feedstuff columns of Table 1 for a specific crop, does not necessarily mean that such items are not produced from this crop, and/or used as human foods or feedstuffs. The PMRA may add and update the table during ongoing assessments of other/novel food and/or feed fractions.

**Table 1. Raw agricultural and processed commodities and feedstuffs derived from field crops**

| Crop | RAC (1) | Processed Commodity (2) | Feed | |
|---|---|---|---|---|
| | | | Feedstuff | %DM (3) |
| Alfalfa (4) | forage<br>hay<br>seed (5) | | forage | 35 |
| | | | hay | 89 |
| | | | meal (6) | 89 |
| | | | silage (7) | 40 |
| Almond | nutmeat and hulls | | hulls | 90 |
| Apple | fruit | pomace, wet juice | pomace, wet | 40 |
| Apricot | fruit (8) | | | |
| Artichoke, Globe | flower head | | | |
| Asparagus | spears (stems) | | | |
| Avocado | fruit (8) | | | |
| Banana (9) | whole fruit | | | |
| Barley (10) | grain (11)<br>hay<br>straw | pearled barley<br>flour<br>bran | grain (11) | 88 |
| | | | hay | 88 |
| | | | straw | 89 |
| Bean (12) | succulent bean seed | | | |
| Beet, garden | root<br>tops (leaves) | | | |
| Beet, sugar | root<br>tops (leaves) | refined sugar(13)<br>dried pulp<br>molasses | tops (leaves)<br>dried pulp | 23<br>88 |
| | | | molasses | 75 |
| Blackberry (14) | berry | | | |
| Blueberry | berry | | | |
| Broccoli | flower head and stem | | | |
| Brussels sprouts | leaf<br>sprouts | | | |
| Buckwheat | grain (15) | flour | | |
| Cabbage | fresh, w/wrapper leaves (16) | | | |
| Cacao bean | bean | roasted bean<br>cocoa powder<br>chocolate | | |
| Canola | seed | meal<br>refined oil | meal | 88 |
| Carob bean | bean | | | |
| Carrot | root | | culls (17) | 12 |

| Crop | RAC (1) | Processed Commodity (2) | Feed | |
|---|---|---|---|---|
| | | | Feedstuff | %DM (3) |
| Cauliflower | flower head and stem | | | |
| Celery | untrimmed leaf stalk (petiole) | | | |
| Cherry, sweet | fruit (8) | | | |
| Cherry, tart (sour) | fruit (8) | | | |
| Chicory | root<br>tops (leaves) | | | |
| Citrus | whole fruit | dried pulp<br>oil<br>juice | dried pulp | 91 |
| Clover (18) | forage<br>hay | | forage | 30 |
| | | | hay | 89 |
| | | | silage (19) | 30 |
| Coconut | coconut (meat and liquid combined) | copra (dried meat)<br>oil | | |
| Coffee (20) | green bean | roasted bean<br>instant coffee | | |
| Collards | greens | | | |
| Corn, field | grain<br>starch (24)<br>forage (21)<br>stover (22)<br>grits<br>flour<br>aspirated grain fractions (23) | wet milling: refined oil<br><br>dry milling: meal and refined oil | grain | 88 |
| | | | forage (21) | 40 |
| | | | stover (22) | 83 |
| | | | aspirated grain fractions (23) | 85 |
| | | | milled byproducts (25) | 85 |
| Corn, pop | grain<br>stover (22) | | grain | 88 |
| | | | stover (23) | 85 |
| Corn, sweet (26) | sweet corn (K+CWHR) (27)<br>forage (28)<br>stover (22) | | forage (28) | 48 |
| | | | stover (22) | 83 |
| | | | cannery waste (29) | 30 |
| Cotton | undelinted seed<br>cottongin bypdts (30) | meal<br>hulls<br>refined oil | undelinted seed | 88 |
| | | | cotton gin byproducts (30) | 90 |
| | | | meals | 89 |
| | | | hulls | 90 |
| Cowpea (31) | seed<br>hay<br>forage | | seed | 88 |
| | | | hay | 86 |
| | | | forage | 30 |
| Crabapple | fruit | | | |
| Cranberry | berry | | | |
| Crownvetch (32) | forage<br>hay | | forage | 30 |
| | | | hay | 90 |
| Cucumber | fruit | | | |
| Currant | fruit | | | |
| Date | dried fruit (8) | | | |
| Dewberry | berry | | | |
| Eggplant | fruit | | | |
| Elderberry | berry | | | |

| Crop | RAC (1) | Processed Commodity (2) | Feed | |
|---|---|---|---|---|
| | | | Feedstuff | %DM (3) |
| Endive/ Escarole | leaves | | | |
| Fig | fruit | dried | | |
| Flax | seed | meal | meal | 88 |
| Garlic | bulb | | | |
| Ginseng | dried root | | | |
| Gooseberry | berry | | | |
| Grape | fruit | raisin<br>juice | | |
| Grass (pasture and rangeland) (33) | forage<br>hay | | forage | 25 |
| | | | hay | 88 |
| | | | silage (34) | 40 |
| Herbs (35) | fresh | dried | | |
| Hops | dried hops cones (36) | | | |
| Horseradish | root | | | |
| Huckleberry | berry | | | |
| Jerusalem artichoke | tuber | | | |
| Kale | leaves | | | |
| Kiwifruit | fruit | | | |
| Kohlrabi | bulbous stem and leaves | | | |
| Kumquat | fruit | | | |
| Leek | whole plant | | | |
| Lentil | seed | | | |
| Lespedeza (37) | forage<br>hay | | forage | 22 |
| | | | hay | 88 |
| Lettuce, head | fresh, w/wrapper leaves (38) | | | |
| Lettuce, leaf | leaves (39) | | | |
| Loganberry | berry | | | |
| Lupin | seed | | seed | 88 |
| Mango | fruit (8) | | | |
| Millet (40) | grain (41)<br>forage<br>hay<br>straw (42) | flour (43) | grain (41) | 88 |
| | | | forage | 30 |
| | | | hay | 85 |
| | | | straw (42) | 90 |
| Mung bean | bean<br>bean sprouts (44) | | | |
| Mushroom | cap and stem | | | |
| Muskmelon (45) | fruit | | | |
| Mustard greens | greens (leaves) | | | |
| Nectarine | fruit (8) | | | |
| Nuts (46) | nutmeat | | | |
| Oats (47) | grain (11)<br>forage<br>hay<br>straw | flour<br>groats/rolled oats | grain (11) | 89 |
| | | | forage | 30 |
| | | | hay | 90 |
| | | | straw | 90 |
| Okra | fruit (pods) | | | |
| Olives | fruit (8) | oil | | |
| Onion, bulb | bulb | | | |
| Onion, green | whole plant w/o roots | | | |
| Papaya | fruit | | | |
| Parsley (48) | leaves, fresh | dried | | |
| Parsnip | root | | | |
| Passion fruit | fruit | | | |

| Crop | RAC (1) | Processed Commodity (2) | Feed | |
|---|---|---|---|---|
| | | | Feedstuff | %DM (3) |
| Pawpaw | fruit | | | |
| Pea (49) | pea, succulent (50)<br>seed (51) | | | |
| Pea, field (52) | seed<br>vines<br>hay | | seed | 90 |
| | | | vines | 25 |
| | | | hay | 88 |
| | | | silage (53) | 40 |
| Peach | fruit (8) | | | |
| Peanut | nutmeat<br>hay (54) | meal<br>refined oil | meal | 85 |
| | | | hay (54)<br>(R) (55) | 85 |
| Pear | fruit | | | |
| Pepper, bell and nonbell (56) | fruit | | | |
| Peppermint | tops (leaves and stems) | oil | | |
| Pimento (57) | fruit | | | |
| Pineapple | fruit | process residue (58)<br>juice | process residue (58) | 25 |
| Plantain (59) | whole fruit | | | |
| Plum | fruit (8) | prune | | |
| Potato | tuber | granules/flakes (60)<br>chips<br>wet peel | Culls | 20 |
| | | | processed potato waste (61) | 12 |
| Pumpkin | fruit | | | |
| Quince | fruit | | | |
| Radicchio (red chicory) | leaves, fresh | | | |
| Radish | root<br>tops (leaves) | | | |
| Rape | seed | meal (62) | meal | 88 |
| | forage | | forage | 30 |
| Rape greens (63) | greens (leaves) | | | |
| Raspberry, black and red | berry | | | |
| Rhubarb | petioles | | | |
| Rice (64) | grain (11)<br>straw | polished rice<br>hulls<br>bran | grain (11) | 88 |
| | | | straw | 90 |
| | | | hulls | 90 |
| | | | bran | 90 |
| Rutabaga | root | | | |
| Rye (65) | grain (66)<br>forage<br>straw | flour<br>bran | grain (66) | 88 |
| | | | forage | 30 |
| | | | straw | 88 |
| Safflower | seed | meal<br>refined oil | meal | 91 |
| Salsify | root<br>tops (leaves) | | | |
| Sesame | seed | oil | | |
| Shallot | bulb | | | |

| Crop | RAC (1) | Processed Commodity (2) | Feed | |
|---|---|---|---|---|
| | | | Feedstuff | %DM (3) |
| Sorghum, grain | grain<br>forage (21)<br>stover (22)<br>aspirated grain fractions (23) | flour (67) | grain | 86 |
| | | | forage (21) | 35 |
| | | | stover (22) | 88 |
| | | | aspirated grain fractions (23) | 85 |
| Sorghum, sweet (68) | stalk | syrup | | |
| Sorghum forages, Sudan grass | (See Grass) | | | |
| Soybean (69) | seed<br>forage<br>hay<br>aspirated grain fractions (23) | meal<br>hulls<br>refined oil | seed | 89 |
| | | | forage (R) (55) | 56 |
| | | | hay (R) (55) | 85 |
| | | | aspirated grain fractions (23) | 85 |
| | | | meal | 92 |
| | | | hulls | 90 |
| | | | silage (70) | 30 |
| Spearmint | tops (leaves and stems) | oil | | |
| Spices (71) | fresh | dried | | |
| Spinach | leaves | | | |
| Squash | fruit | | | |
| Strawberry | berry | | | |
| Sugarcane (72) | cane | molasses (73)<br>refined sugar (13) | molasses (73) | 75 |
| Sunflower | seed | meal<br>refined oil | meal | 92 |
| Sweet potato | root | | | |
| Swiss chard | petioles | | | |
| Taro | corm<br>foliage | | | |
| Tea (74) | plucked leaves | dried<br>instant tea | | |
| Tomato | fruit | paste (75)<br>puree | | |
| Trefoil (76) | forage<br>hay | | forage | 30 |
| | | | hay | 85 |
| Turnip | root<br>tops (leaves) | | root | 15 |
| | | | tops (leaves) | 30 |
| Vetch (77) | forage hay | | forage | 30 |
| | | | hay | 85 |
| Watercress | leaves and stems | | | |
| Watermelon | fruit | | | |
| Wheat (78)<br>(79) | grain (66)<br>forage<br>hay<br>straw<br>aspirated grain fractions (23) | bran<br>flour<br>middlings<br>shorts<br>germ | grain (66) | 89 |
| | | | forage | 25 |
| | | | hay | 88 |
| | | | aspirated grain fractions (23) | 85 |
| | | | milled byproducts (80) | 88 |
| Yam | tuber | | | |

**Table notes**. The following notes are referenced in the table.

1) **RAC**. Raw agricultural commodity.
2) **Processed Commodity.** A wide range of raw agricultural commodities are processed before they are consumed by humans or as animal feedstuffs.
3) **% DM (percent dry matter).** For beef and dairy feedstuffs, the percent moisture should be reported for representative samples of raw agricultural and processed commodities.
4) **Alfalfa.** Residue data are needed from a minimum of three cuttings unless climatic conditions restrict the number of cuttings. Cut sample at late bud to early bloom stage (first cut), and/or at early (one tenth) bloom stage (later cuts).
5) **Alfalfa seed.** For registered uses on alfalfa grown for seed, residue data should be provided on seed, forage and hay; in order to allow the feeding of seed screenings and aftermath to livestock.
6) **Alfalfa meal.** Residue data are not needed for meal; however, the meal should be included in the livestock diet, using the hay residue level. Hay should be field dried to a moisture content of 10 to 20 percent.
7) **Alfalfa silage.** Residue data on silage are optional, but are desirable for assessment of dietary burden. Cut at late bud to one-tenth bloom stage for alfalfa, allow to wilt to approximately 60 percent moisture, then chop fine, pack tight, and allow to ferment for three weeks maximum in an airtight environment until it reaches pH 4. This applies to both silage and haylage. In the absence of silage data, residues in forage will be used for silage, with correction for DM.
8) **Fruit.** Fruit should be analyzed after removing and discarding the stem, and stone or pit.
9) **Banana**. Field residue data on both bagged and unbagged bananas should be provided. The required number of field trials may be split between bagged and unbagged bananas. Alternatively, one sample each of bagged and unbagged bananas may be taken from each site. Data are required on the whole commodity, including peel after removing and discarding the crown tissue and stalk. At the applicant's discretion, residue data on just the banana pulp may be provided for purposes of dietary risk assessment.
10) **Barley hay.** Cut when the grain is in the milk to soft dough stage. Hay should be field dried to a moisture content of 10 to 20 percent. **Barley straw.** Plant residue (in other words, dried stalks or stems with leaves), left after the grain has been harvested (in other words, threshed). **Barley silage**. Residue data on silage are optional, but are desirable for assessment of dietary exposure. Cut sample at boot to early head stage, allow to wilt to 55 to 65 percent moisture, then chop fine, pack tight, and allow to ferment for three weeks maximum in an air-tight environment until it reaches pH 4. In the absence of silage data, residues in forage will be used for silage, with correction for dry matter.
11) **Barley grain, oat grain, or rice grain.** Kernel (caryopsis) plus hull (lemma and palea).
12) **Bean.** See Crop Group 6: *Legume Vegetables* for cultivars of beans. **Bean seed.** Dried seed for uses on dried shelled beans; succulent seed without pod for uses on succulent shelled beans (for example, lima beans); succulent seed with pod for edible-podded beans (for example, snap beans). Cowpea is the only bean crop

considered for livestock feeding. See cowpea. Residue data for forage and hay are required only for cowpea.

13) **Beet, sugar.** Residue data may be provided for raw sugar or refined sugar, or both raw and refined.
14) **Sugarcane.** Residue data may be provided in the same manner.
15) **Blackberry.** See Crop Group 13-07: *Berries* for cultivars of blackberries.
16) **Buckwheat grain.** Seed (achene) plus hull.
17) **Cabbage fresh, with wrapper leaves.** Entire cabbage head with obviously decomposed or withered leaves removed. In addition, residue data on cabbage head, without wrapper leaves, are desirable particularly when a more accurate assessment of dietary exposure is necessary.
18) **Carrot culls.** Data for raw agricultural commodities will cover residues on culls.
19) **Clover forage.** Cut sample at the 10 to 20 centimeters to prebloom stage, at approximately 30 percent DM. **Clover hay.** Cut at early to full bloom stage. Hay should be field dried to a moisture content of 10 to 20 percent. Residue data for clover seeds are not needed.
20) **Clover silage.** Residue data on silage are optional, but are desirable for assessment of dietary exposure. Cut sample at early to one-fourth bloom stage for clover, allow to wilt to approximately 60 percent moisture, then chop fine, pack tight, and allow to ferment for three weeks maximum in an airtight environment until it reaches pH 4. This applies to both silage and haylage. In the absence of silage data, residues in forage will be used for silage, with correction for DM.
21) **Coffee.** Residue data are required on the green bean, the roasted bean, and on instant coffee. MRLs on the roasted bean and instant coffee will be established, if residues exceed those on the green bean. The green bean is the dried seed of the coffee bean.
22) **Corn forage (field and pop).** Cut sample, in other words, whole aerial portion of the plant, at late dough/early dent stage (black ring/layer stage for corn only). **Sorghum forage.** Cut sample, in other words, whole aerial portion of the plant, at soft dough to hard dough stage. Forage samples should be analyzed as is, or may be analyzed after ensiling for three weeks maximum, and reaching pH 5 or less, with correction for DM.
23) **Corn stover (filed and pop).** Mature dried stalks from which the grain or whole ear, in other words, cob + grain, has been removed; containing 80 to 85 percent DM. **Sorghum stover.** Mature dried stalks from which the grain has been removed; containing approximately 85 percent DM.
24) **Aspirated grain fractions**, previously called **grain dust**. Dust collected at grain elevators for environmental and safety reasons. Residue data should be provided for any postharvest use on corn, sorghum, soybeans, or wheat. For a preharvest use after the reproduction stage begins and seed heads are formed, data are needed unless residues in the grain are less than the limit of quantitation (LOQ) of the analytical method. For a preharvest use during the vegetative stage, in other words, 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, for example, wheat bran or soybean hulls.
25) **Corn starch.** Residue data from starch will be used for corn syrup. Applicants may also provide data on syrup for a more accurate assessment of dietary exposure.

26) **Corn milled byproducts.** Use residue data for corn dry-milled processed commodities (grits, meal, and flour) having the highest residues, excluding oils.
27) **Sweet corn.** Residue data on early sampled field corn should suffice to provide residue data on sweet corn, provided that the residue data are generated at the milk stage on kernel plus cob with husk removed, and there are adequate numbers of trials and geographical representation from the sweet corn growing regions.
28) **Sweet corn (K + CWHR)**. Kernels plus cob with husks removed.
29) **Sweet corn forage.** Samples should be taken when sweet corn is normally harvested for fresh market, and may or may not include the ears. Applicants may analyze the freshly cut samples, or may analyze the ensiled samples after ensiling for three weeks maximum, and reaching pH 5 or less, with correction for percent DM.
30) **Sweet corn cannery waste.** Includes husks, leaves, cobs, and kernels. Residue data for forage will be used for sweet corn cannery waste.
31) **Cotton gin byproducts,** commonly called **gin trash**. Includes the plant residues from ginning cotton, and consists of burrs, leaves, stems, lint, immature seeds, and sand and/or dirt. Cotton must be harvested by commercial equipment, in other words, stripper and mechanical picker, to provide an adequate representation of plant residue for the ginning process. At least three field trials for each type of harvesting, in other words, stripper and picker, are needed, for a total of six field trials.
32) **Cowpea forage.** Cut forage at 15 centimeters to prebloom stage, at approximately 30 percent DM. **Cowpea hay.** Cut when pods are one-half to fully mature. Hay should be field dried to a moisture content of 10 to 20 percent.
33) **Crownvetch forage.** Cut sample at 15 centimeters to prebloom stage, at approximately 30 percent DM. **Crown vetch hay.** Cut at full bloom stage. Hay should be field dried to a moisture content of 10 to 20 percent.
34) **Grass.** Zero-day crop field residue data for grasses cut for forage should be provided unless it is not feasible, for example, preplant/preemergent pesticide uses. A reasonable interval before cutting for hay is allowed. **Grass forage.** Cut sample at 15 to 20 centimeters to boot stage, at approximately 25 percent DM. **Grass hay.** Cut in boot to early head stage. Hay should be field dried to a moisture content of 10 to 20 percent. Grasses include barnyard grass, bent grass, Bermuda grass, Kentucky bluegrass, big bluestem, smooth bromegrass, buffalograss, reed canarygrass, crabgrass, cupgrass, dallisgrass, sand dropseed, meadow foxtail, eastern gamagrass, side-oats grama, guinea grass, Indian grass, Johnson grass, love grass, napier grass, oat grass, orchardgrass, pangola grass, redtop, Italian ryegrass, sprangletop, squirrel-tail grass, stargrass, switchgrass, timothy, crested wheatgrass, and wild ryegrass. Also included are Sudan grass and sorghum forages and their hybrids. For grass grown for seed only, pregrazing intervals and preharvest intervals are acceptable. Residue data may be based on the regrowth after harvesting the seed.
35) **Grass silage.** Residue data on silage are optional, but are desirable for the assessment of dietary burden. Cut sample at boot to early head stage, allow to wilt to 55 to 65 percent moisture, then chop fine, pack tight, and allow to ferment for three weeks maximum in an airtight environment until it reaches pH 4. In the absence of silage data, residues in forage will be used for silage, with correction for DM.
36) **Herbs.** Consist primarily of leaves, stems, and flowers and are marketed fresh, in other words, succulent, or dried.

37) **Hops, cones, dried.** Dried hops will be considered as a raw agricultural commodity for regulatory purposes. Residue data are needed for dried hops only.
38) **Lespedeza forage.** Cut sample at 10 to 15 centimeters to prebloom stage, at 20 to 25 percent DM. **Lespedeza hay: Annual/Korean.** Cut at early blossom to full bloom stage. **Sericea.** Cut when 30 to 38 centimeters tall. Hay should be field dried to a moisture content of 10 to 20 percent.
39) **Lettuce, fresh, with wrapper leaves.** Entire lettuce head, with the obviously decomposed or withered leaves removed. In addition, residue data on lettuce head, without wrapper leaves, are desirable, particularly when more accurate assessment of dietary exposure is necessary.
40) **Lettuce, leaf.** Residue data should be on samples with the obviously decomposed or withered leaves removed.
41) **Millet forage.** Cut sample at 25 centimeters to early boot stage, at approximately 30 percent DM. **Millet hay.** Cut at early boot stage or approximately 102 centimeters tall, whichever is reached first. Hay should be field dried to a moisture content of 10 to 20 percent. Millet, including pearl millet.
42) **Millet grain.** Kernel plus hull (lemma and palea). **Pearl millet grain.** Kernel with hull (lemma and palea) removed.
43) **Millet straw.** Data are required for proso millet only. **Proso millet straw.** Plant residue, in other words, dried stalks or stems with leaves, left after the grain has been harvested.
44) **Millet flour.** Not produced significantly for human consumption. Residue data are not needed at this time.
45) **Mung bean**. Data on mung bean covers sprouts except when the pesticide is used on the sprouts per se.
46) **Muskmelon.** Includes cantaloupe, casaba, crenshaw, etc. See Crop Group 9: Cucurbit Vegetables
47) **Nuts.** Includes Croup Group 14-11: *Tree Nuts*, except almonds. Almond hulls are considered a significant feedstuff. Hulls from other tree nuts are not considered significant feedstuffs.
48) **Oats forage.** Cut sample between tillering to stem elongation (jointing) stage. **Oats hay.** Cut sample from early flower to soft dough stage. Hay should be field dried to a moisture content of 10 to 20 percent. **Oats straw.** Cut plant residue, in other words, dried stalks or stems with leaves, left after the grain has been harvested, in other words, threshed.
49) **Parsley.** Fresh parsley is included in Crop Subgroup 4-13A Leafy Greens. Dried parsley is included in Crop Subgroup 25B Herbs Dried Leaves
50) **Pea.** Residue data for forage and hay are required for cowpea. See cowpea. Residue data for vines and hay are required for field peas only. See pea, field.
51) **Pea, succulent.** Succulent seed with pod for edible-podded peas, for example, snow peas; succulent seed without pod for uses on succulent shelled peas, for example, English peas.
52) **Pea seed.** Mature dried seed for uses on dried, shelled peas.
53) **Pea, field.** Does not include the canning field pea cultivars used for human food. Includes cultivars grown for livestock feeding only, such as **Australian winter pea. Field pea vines.** Cut sample any time after pods begin to form, at approximately 25 percent DM. **Field pea hay.** Succulent plant cut from full bloom through pod formation. Hay should be field dried to a moisture content of 10 to 20 percent.

54) **Pea, field, silage.** Use field pea vine residue data for field pea silage with correction for DM.
55) **Peanut hay.** Peanut hay consists of the dried vines and leaves left after the mechanical harvesting of peanuts from vines that have been sun-dried to a moisture content of 10 to 20 percent.
56) **(R):** Label restrictions against feeding may be allowed, for example, "Do not feed green immature growing plants to livestock, or, Do not harvest for livestock feed."
57) **Pepper.** Non-bell pepper includes chili pepper.
58) **Pimento.** The official name adopted by the Georgia Pimento Growers Association.
59) **Pineapple process residue,** also known as **wet bran**. A wet waste byproduct from the fresh-cut product line that includes pineapple tops (minus crown), bottoms, peels, any trimmings with peel cut up, and the pulp that is left after squeezing for juice; it can include culls.
60) **Plantain.** Banana MRL will cover plantain.
61) **Potato granules/flakes.** Residue data may be provided for either.
62) **Processed potato waste.** MRLs for wet peel should be used for dietary burden calculations. Residue data may be provided from actual processed potato waste generated, using a pilot or commercial scale process that gives the highest percentage of wet peel in the waste.
63) **Rapeseed meal.** Residue data are not needed for rapeseed oil since it is produced for industrial uses and is not an edible oil. The edible oil is only produced from canola. See canola.
64) **Rape greens.** A commodity listed in Crop Subgroup 4-13B *Brassica* Leafy Greens
65) **Rice straw.** Stubble, in other words, basal portion of the stems, left standing after harvesting the grain.
66) **Rye forage.** Cut sample at 15 to 20 centimeters stage to stem elongation (jointing) stage, at approximately 30 percent DM. **Rye straw.** Cut plant residue, in other words, dried stalks or stems with leaves, left after the grain has been harvested, in other words, threshed.
67) **Rye grain, triticale or wheat grain.** Kernel (caryopsis) with hull (lemma and palea) removed.
68) **Sorghum flour.** Residue data are not needed at this time since sorghum flour is used exclusively in the United States as a component for drywall, and not as either a human food or a feedstuff. However, because 50 percent of the worldwide sorghum production goes toward human consumption, data may be needed at a later date.
69) **Sorghum, sweet.** Sweet sorghum commodities, in other words, seed and forage.
70) **Soybean forage.** Cut samples at 15 to 20 centimeters tall (sixth node) to beginning pod formation, at approximately 35 percent DM. **Soybean hay.** Cut samples at mid-to-full bloom stage and before bottom leaves begin to fall, or when pods are approximately 50 percent developed. Hay should be field dried to a moisture content of 10 to 20 percent.
71) **Soybean silage.** Residue data on silage are optional. Harvest sample when pods are one-half to fully mature, in other words, full pod, stage. In the absence of silage data, residues in forage will be used for silage, with correction for DM.
72) **Spices.** Include aromatic seeds, buds, bark, berries, pods, and roots consumed and marketed primarily in their dried form. See Crop Group 26 Spices for a listing of spices.

73) **Sugarcane bagasse.** Information indicates that sugercane bagasse is mainly used for fuel. Residue data will not be needed at this time, but may be needed at a later date.
74) **Sugarcane molasses.** Residue data are needed for blackstrap molasses.
75) **Tea.** Residue data are required on plucked or freshly picked leaves, dried tea, and instant tea.
76) **Tomato paste.** Residue data on tomato paste cover tomato processed products, for example, sauce, juice and catsup, except tomato puree, which covers canned tomatoes.
77) **Trefoil forage.** Cut sample at 13 to 25 centimeters or early bloom stage, at approximately 30 percent DM. **Trefoil hay.** Cut at first flower to full bloom. Hay should be field dried to a moisture content of 10 to 20 percent.
78) **Vetch forage.** Cut sample at 15 centimeters to prebloom stage, at approximately 30 percent DM. **Vetch hay.** Cut at early bloom stage to when seeds in the lower half of the plant are approximately 50 percent developed. Hay should be field dried to a moisture content of 10 to 20 percent. Vetch does not include crownvetch.
79) **Wheat forage.** Cut sample at 15 to 20 centimeters stage to stem elongation (jointing) stage, at approximately 25 percent DM. **Wheat hay.** Cut samples at early flower (boot) to soft dough stage. Hay should be field dried to a moisture content of 10 to 20 percent. **Wheat straw.** Cut plant residue, in other words, dried stalks or stems with leaves, left after the grain has been harvested, in other words, threshed.
80) **Wheat.** Includes emmer wheat and triticale. No processing study is needed for a specific MRL on emmer wheat.
81) **Wheat milled byproducts.** Use highest value for wheat middlings, bran, and shorts

**Reference**

USEPA (2020). Title 40: Protection of Environment Part 180 – Tolerances and Exemptions for Pesticide Chemical Residues in Food Subpart B – Procedural Regulations. https://www.ecfr.gov/cgi-bin/text-idx?SID=46e0f15c31b8f5b1c0a348eac9f380e5&mc=true&node=se40.24.180_141&rgn=div8

# Appendix II

## Description of crop field trial regions

This Appendix describes the seven major and the four minor field trial regions. Each of these regions recognizes physical characteristics, such as soils, and crops and climate that make the region unique within the Canadian agricultural landscape. The subzones address differences within a region, generally reflected in the types of crops grown in that region. The Canadian regions, as much as possible, correspond to the US regions.

The **Appalachian zone (Zone 1)**, extends throughout New Brunswick, Gaspé, and the Appalachian Region of Southern Québec. Humo-Ferric Podzols dominate this region with pockets of Gray Luvisols and Dystric Brunisols. These marginal to intermediate soils exist in an area of considerable relief, especially in mountainous areas of the Appalachians. A humid temperate climate exists in this region with low to intermediate values for most climatic indicators, such as corn heat units. In general, this region contains marginal agricultural capability with pockets of intermediate capability. Potatoes, grains, tame hay, and limited vegetable crops comprise the agriculture in this region.

A more favourable growing climate in the **Atlantic zone (Zone 1A)**, distinguishes it from Zone 1. As a result, this region has a greater potential for fruit and vegetable production compared to Zone 1.

The **Southern Ontario zone (Zone 5)**, extends from Windsor to the St. Lawrence River, just north of Kingston. Grey Brown Luvisols dominate this region with considerable areas of Humic Gleysols in the south and Melanic Brunisols in the north. These good soils result in high land capability ratings for agriculture under the Canada Land Inventory. The region experiences a moderate climate favourable to a wide variety of crops. Predominantly flat terrain is found in this region with some regional anomalies, such as the Niagara Escarpment. This zone is characterized by Canada's most diverse mix of crops, including extensive fruit, vegetable, grain, and corn production.

Zone 5 also includes a small portion of Southern Manitoba. Although the soils and climate of this Manitoba region differ from Southern Ontario, it is included with Zone 5 in order to maintain integrity with the American delineation. Also, certain crops, notably corn, are found in both regions. However, in contrast to Southern Ontario, soils in this region are predominately Black Chernozemic on flat terrain. The climate is much drier than Southern Ontario, as indicated by the Dry Subhumid Thornthwaite Classification.

The **Northern Shield zone (Zone 5A)**, differs from both the St. Lawrence Valley and Southern Ontario zones, but in keeping with the American classification, remains related to those two zones. This region includes the various pockets of agriculture that extend from Manitoba to the north shore of the Gulf of St. Lawrence. The island of Newfoundland is also included. This region of the Canadian Shield is dominated by rough to rolling terrain of HumoFerric Podzols of marginal to poor agricultural capability. Agricultural activity occurs in pockets of Brunisols and Gleysols found throughout this region. Climatic

conditions vary considerably throughout this region, but are generally not conducive to agricultural activity. Tame hay, grains, and some specialty fruits are the main crops of this region. Very little agricultural activity is found in Newfoundland. The island's topography, mountainous in some areas, is generally unsuitable for agriculture. The soils, climate, topography, and limited agriculture distinguish this region from Nova Scotia and New York State in United States Zone 1.

The **St. Lawrence Valley zone (Zone 5B)**, produces fruits, vegetables and corn similar to the crops of Southern Ontario. Climatological and soil characteristics are the main criteria for the boundary separating Zones 5 and 5B. The Montréal area is dominated by Melanic Brunisols of good agricultural capability. Down river, the soils change to Humic Gleysols and Humo-Ferric Podzols of marginal agricultural capability. Shorter frost-free periods and lower corn heat units are the main climatic differences that distinguish this region from Zone 5.

The **Dryland Prairie zone (Zone 7)**, extends from west of Regina to near the Alberta border. Brown and Dark Brown Chernozemic soils dominate this region with pockets of Brown Solonetz and Orthic Regosol soils. Agricultural capability remains good with some limitations associated with a dry climate. The Thornthwaite Classification rates this region as semiarid to dry subhumid. Other key climatic indicators, such as growing degree days, indicate generally favourable growing conditions with minor limitations. Topography is flat with pockets of rolling terrain. Grains and hay are the main crops produced in this zone, with a lack of the specialty crops found in Subzone 7A, Zone 5, Manitoba, or Zone 14.

The **Southern Alberta Irrigation zone (Zone 7A)**, is an anomalous zone due to irrigation practices. All datasets, including soil and climatic variables, indicate that this region should be included with Zone 7. However, as a result of irrigation, several vegetable crops are found in this region, including sweet corn, green peas, sugar beets, potatoes and cucumbers.

The **Rocky Mountains zone (Zone 9)**, includes the moist region of the eastern reaches of the Canadian Rockies. Topography is hilly to mountainous with a variety of soils. The climate varies considerably, with Thornthwaite classes ranging from humid to dry subhumid. This region lacks the large, isolated areas of agricultural activity found in the United States. In Canada, small pockets of agriculture can be found in the southern portions of this zone. These areas of agriculture cannot be considered statistically valid for determining trends in crops for this zone.

The **Dryland Interior zone (Zone 11)**, encompasses the dryland interior of British Columbia. Mountainous topography limits agricultural activity to valleys. Considerable crop variation arises due to local topography, soils, and climate. Dark Brown and Dark Grey Chernozemic are the main soils in this region. Climate is dry subhumid to semiarid, but variables, such as sunlight hours, favour agriculture in these areas. Crops vary considerably in this region, from tame hay and grains in the Kamloops area to a variety of fruit and vegetable crops in the Okanagan Valley.

The **Pacific zone (Zone 12)**, includes the wet coastal regions of British Columbia and Vancouver Island. Agriculture is limited to the Fraser Valley and pockets on Vancouver

Island. A Humic Gleysol soil characterizes the Fraser Valley, and Dystric Brunisols are found on Vancouver Island. Mountainous topography is a major limitation for agriculture in this region; however, the warm wet climate favours agricultural activity.

The **Northern Prairie zone (Zone 14)**, covers the Northern Prairies from Manitoba to the Peace River area of Northern Alberta. Agricultural activity is found primarily in Black Chernozemic and Luvisolic soils. This zone includes the Dark Brown Chernozems of Zone 7, and the Solonetzic soils of the Peace River area. Agricultural capability indicators in this region vary from excellent in the interior to marginal along the fringes, with non-agricultural soils in the Boreal Forest regions. Zone 14 experiences a predominantly dry subhumid continental climate; however, other climatological indicators, for example, corn heat units and growing degree days, vary throughout the region. Crop production varies throughout this zone. Canola, barley, peas, and mustard seed are the crops that differentiate this zone from Zone 7.

**Geographic descriptions for safe zones within crop field trial regions**

This subsection describes the field trial regions as geographic safe zones. Within each complex field trial region, simplified polygons have been delineated to provide latitude/longitude coordinates and/or easily recognizable physical or administrative features. The safe zones are considered as areas that are unequivocally within a given field trial region. Refer to Appendix 4 for maps defining Canadian major and minor crop field trial regions.

**Locating a trial site in a zone/region:**

Maps defining safe zone areas in major and minor crop field trial regions of each province of Canada are presented in Appendix 4. These maps are used alongside reference maps[4] to locate crop field trial sites in a safe zone. Reference maps showing crop regions/zones overlayed on the Census Division maps are presented in Appendix 4. The applicant may utilize these maps to assign a trial site to a given region/zone and may confirm its location in a safe zone by referring to the longitudinal/latitudinal coordinates listed below. In addition, the applicant may utilize hand-held or other GPS monitors (Global or Geo Postioning System) to identify coordinates of their trial sites.

Crop field trials conducted in a transition zone may generate residue data that may or may not reflect the residue pattern seen in the safe zones. Therefore, the acceptability of the residue data from transition zones is assessed during the evaluation of the residue data.

The applicant must indicate the location of trial sites on the maps presented in Appendix 4 and provide longitudinal and latitudinal coordinates for each site.

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[4] Geographical Classification SGC 1991 Volume II, published by authority of the Minister responsible for Statistics Canada,
Catalogue 12-572, ISBN 0-660-56558-7. Other maps may be used, e.g., topographical maps.

**Appalachian (Zone 1)**

The safe zone includes all of the province of New Brunswick and most of the south shore of Québec, including all of the Gaspé Peninsula. The western boundary of the zone runs from the south shore of the St. Lawrence River at geographic map coordinate **(1) 47"" 8' 31"N & 70""20' 56"W to (2) 46"" 50' 56"N & 70"" 25' 55"W, (3) 46"" 31' 44"N & 71"" 11' 42"W, 4) 46"" 1' 16"N & 71"" 12' 50"W**. It meets the Québec/Vermont border at **(5) 44"" 59' 17"N & 72"" 36' 47"W**. The southern boundary follows the Québec/Vermont, Québec/New Hampshire, Québec/Maine and New Brunswick/Maine border. The northern boundary of the safe zone follows the south shore of the St. Lawrence River around the Gaspé Peninsula and Baie des Chaleurs.

**Atlantic (Zone 1A)**

The safe zone includes all of Nova Scotia, including Cape Breton Island, and Prince Edward Island.

**Southern Ontario (Zone 5)**

The Southern Ontario safe zone is split between two safe zone polygons. One of the polygons is located in Southern Manitoba south of Winnipeg. The other polygon includes most of Southern Ontario. It also includes Pelee Island in Lake Erie and Manitoulin Island in Georgian Bay.

The southern boundary of the safe polygon in Manitoba runs along the Canada/United States border at **(1) 49"" 00' 00"N & 98"" 5' 49"W to (2) 49"" 00' 00"N & 96"" 46' 12"W**. The polygon is also bounded by **(3) 49"" 55' 44"N & 98"" 21' 50"W** and **(4) 50"" 8' 46"N & 96"" 46' 55"W**.

The northern boundary of the safe zone polygon in Southern Ontario runs from Georgian Bay at **(1) 44"" 29' 10"N & 80"" 3' 32"W** to the St. Lawrence River at **(2) 44"" 13' 1"N & 76"" 28' 8"W**. All regions of Southern Ontario south of this line, including the Bruce Peninsula, are also included.

**Northern Shield (Zone 5A)**

The safe zone polygon includes large areas of Northern Ontario, Northern Québec, the island of Newfoundland and Anticosti Island. The safe zone also includes a small portion of Eastern Manitoba and the southern tip of Labrador.

The polygon follows the Ontario/Minnesota border beginning at **(1) 48"" 33' 58"N & 93"" 52' 12"W** where it meets the north shore of Lake Superior. The southern boundary of the polygon follows the north shore of Lake Superior, and the shoreline of North Channel and Georgian Bay. The polygon extends eastward from the shoreline of Georgian Bay at **(2) 44"" 59' 35"N & 79"" 58' 55"W** to **(3) 44"" 31' 26"N & 76"" 32' 10"W**. The polygon runs northwards where it meets the Ottawa River at **(4) 45"" 40' 5"N & 76"" 37' 26"W**. The boundary continues eastward through Québec where it reaches the north shoreline of the St. Lawrence

River at (**5) 47"" 39' 29"N & 70"" 6' 7"W**. The polygon follows the north shoreline of the St. Lawrence River until it reaches the northern shore of the Gulf of St. Lawrence (just east of the Labrador border) at **(6) 51"" 43' 12"N & 56"" 28' 55"W**. The northern boundary of the polygon runs westward across Northern Québec and Northern Ontario through **(7) 51""**

**18' 29"N & 64"" 24' 14"W, (8) 51"" 4' 55"N & 70"" 3' 4"W, (9) 50"" 22' 1"N & 70"" 18' 54"W, (10) 49"" 57' 11"N & 74"" 13' 48"W, (11) 51"" 43' 19"N & 74"" 0' 14"W, (12) 51"" 45' 36"N & 76"" 6' 47"W, (13) 49"" 2' 56"N & 77"" 52' 55"W, (14) 49"" 30' 4"N & 84"" 12' 25"W** to the extreme western boundary at **(15) 52"" 1' 23"N & 95"" 41' 27"W**. The western boundary runs southward to **(16) 50"" 14' 6"N & 95"" 42' 32"W** and runs eastwards to meet **(17) 50"" 13' 34"N & 93"" 50' 46"W**. The polygon closes at the Ontario/Minnesota border at **(1) 48"" 33' 58"N & 93"" 52' 12"W**.

**St. Lawrence Valley (Zone 5B)**

The safe zone polygon includes the parts of Eastern Ontario, the Montréal region and parts of the North Shore and Eastern Townships of Québec. The polygon is bounded by geographic map coordinates **(1) 45"" 6' 29"N & 74"" 49' 8"W, (2) 45"" 5' 53"N & 73"" 2' 24"W, (3) 46"" 8' 2"N & 71"" 51' 14"W, (4) 46"" 42' 29"N & 71"" 52' 37"W, (5) 45"" 36' 25"N & 74"" 32' 28"W, (6) 45"" 15' 29"N & 76"" 4' 30"W** and **(7) 44"" 38' 46"N & 75"" 46' 8"W**.

**Dryland Prairie (Zone 7**)

The safe zone polygon is located in Alberta and Saskatchewan. The southern boundary of the polygon runs along the Canada/United States border between **(1) 49"" 00' 00"N & 110"" 41' 46"W** and **(2) 49"" 00' 00"N & 102"" 58' 41"W**. The polygon is also bounded by geographic map coordinates in latitude and longitude as follows: **(3) 50"" 54' 29"N & 105"" 12' 58" W, (4) 51"" 36' 18"N & 104"" 40' 12"W, (5) 52"" 5' 38"N & 107"" 28' 30"W, (6) 51"" 36' 18"N & 107"" 55' 37"W, (7) 51"" 39' 40"N & 111"" 8' 46"W, (8) 50"" 31' 55"N & 112"" 4' 5"W, (9) 50"" 22' 52"N & 110"" 12' 18"W, (10) 49"" 42' 11"N & 109"" 50' 49"W** and **(11) 49"" 37' 41"N & 110"" 36' 00"W**.

**Southern Alberta (Zone 7A)**

The safe zone polygon lies entirely within Alberta. The southern boundary of the polygon runs along the Canada/United States border between (**1) 49"" 00' 00"N & 112"" 12' 40"W** and **(2) 111"" 7' 26"W**. The polygon is also bounded by geographic map coordinates in latitude and longitude as follows: **(3) 49"" 57' 50"N & 110"" 59' 42"W** and **(4) 50"" 2' 31"N & 112"" 22' 8"W**.

**Rocky Mountains (Zone 9)**

The safe zone polygon falls on both sides of the Alberta and British Columbia border. The southern boundary of the polygon runs along the Canada/US border between **(1) 49"" 00' 00"N & 115"" 54' 47"W** and **(2) 49"" 00' 00"N & 114"" 24' 14"W**. The polygon is also bounded by geographic map coordinates in latitude and longitude as follows: (**3) 51"" 37' 48"N & 115"" 37' 1"W, (4) 54"" 54' 22"N & 121"" 13' 34"N, (4) 54"" 54' 22"N & 122"" 23' 38"W** and, **(5) 51"" 31' 1"N & 116"" 24' 25"W**.

**Dryland Interior (Zone 11)**

The safe zone polygon lies entirely within mainland British Columbia. The southern boundary of the polygon runs along the Canada/United States border between **(1) 49"" 00' 0"N & 121"" 1' 55"W** and **(2) 49"" 00' 0"N & 117"" 20' 24"W**. The polygon is also bounded by geographic map coordinates in latitude and longitude as follows: **(3) 51"" 16' 55"N & 117"" 28' 34"W**, (4) **53"" 16' 41"N & 121"" 34' 48"W, (5) 55"" 34' 26"N & 124"" 21' 58"W, (6) 54"" 15' 25"N & 126"" 37' 26"W** and, **(7) 51"" 1' 8"N & 121"" 52' 52"W**.

**Pacific (Zone 12)**

Includes the Queen Charlotte Islands, Vancouver Island and the Gulf Islands of British Columbia. The polygon is also bounded by geographic map coordinates in latitude and longitude as follows: **(1) 55"" 25' 8"N & 127"" 44' 42"W, (2) 53"" 34' 26"N & 127"" 48' 00"W, (3) 51"" 51" 39"N & 125"" 34' 48"N** and, **(4) 50"" 13' 23"N & 121"" 57' 22"W**. The west border of the safe zone is bounded by the west coast of mainland British Columbia and begins in the south at the US border (at **49"" N**) running north along the coast to **129"" 40' 8"N & 54"" 57' 7"W**. To the east, the safe zone follows the Canada/US border to **121"" 57' 22"W**.

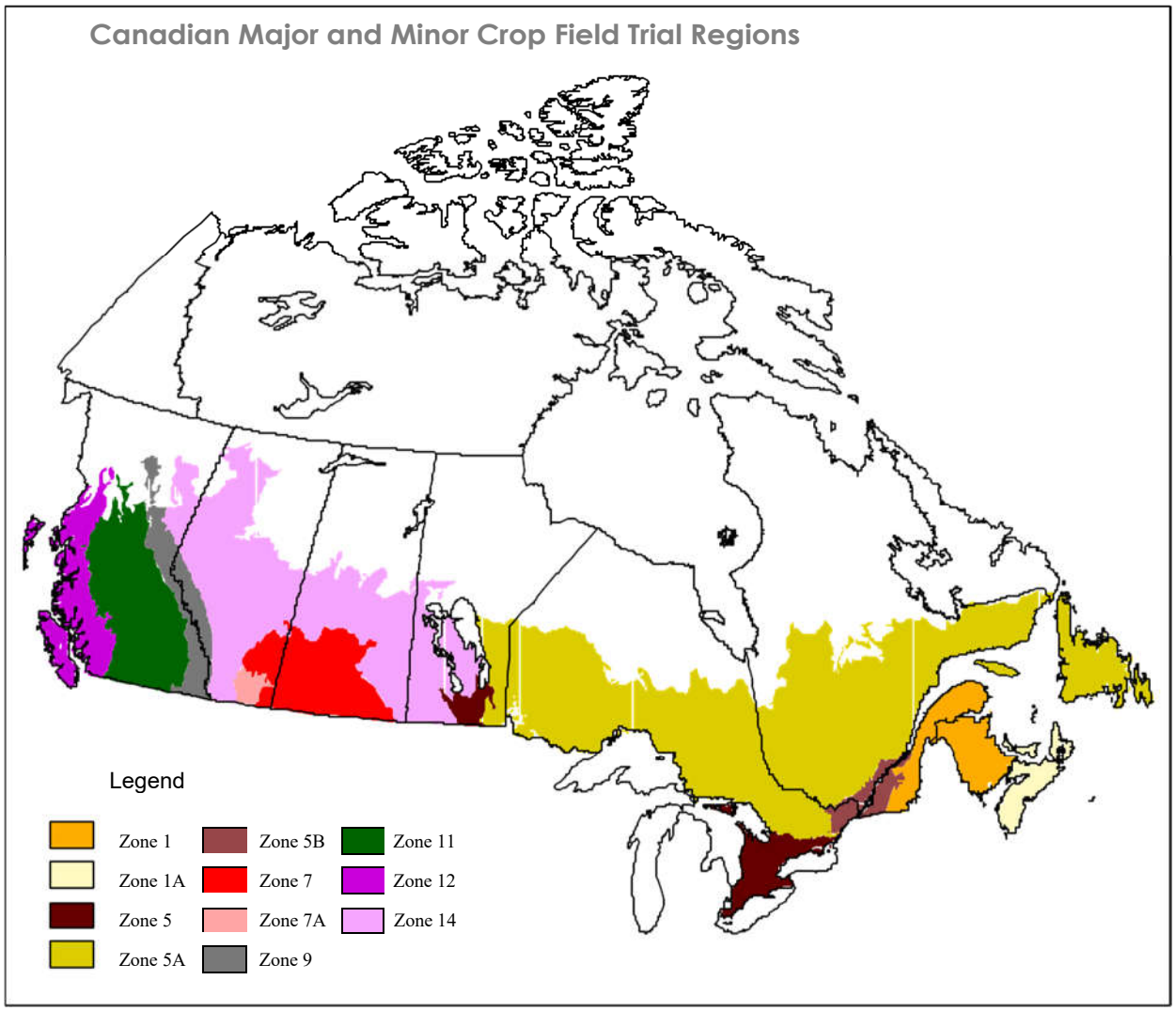
**Northern Prairie (Zone 14)**

The Northern Prairie (Zone 14) is split between two safe zone polygons. One of these polygons is located entirely within Manitoba, within the inter-lake district between Lake Winnipeg, Lake Winnipegosis, Lake Manitoba and Dauphin Lake. The other safe zone polygon is located in regions of Alberta, Saskatchewan and Manitoba. The geographic map coordinates of the safe polygon that is located entirely within Manitoba are expressed in latitude and longitude as follows: **(1) 53"" 3' 7"N & 99"" 12' 58"W, (2) 51"" 48' 32"N & 98"" 4' 37"W, (3) 51"" 47' 24"N & 97"" 27' 54"W, (4) 50"" 26' 6"N & 97 "" 13' 12"W, (5) 50"" 14' 13"N & 97"" 47' 38"W** and **(6) 52"" 46' 44"N & 99"" 36' 40"W**.

The other safe zone polygon, the polygon that is located in portions of Alberta, Saskatchewan and Manitoba, runs along the Canada/US border in Alberta between **(1) 49"" 00' 00"N & 112"" 35' 49"W** and **(2) 49"" 00' 00"N & 113"" 37' 41"W**. This polygon also runs along the Saskatchewan and Manitoba Canada/United States border between **(3) 49"" 00' 00"N & 99"" 32' 24"W** and **(4) 49"" 00' 00"N & 101"" 54' 11"W**. The polygon is also bounded by **(5) 49"" 43' 44"N & 102"" 24' 40"W, (6) 51"" 8' 28"N & 104"" 23' 17"W, (7) 52"" 2' 42"N & 103"" 35' 49"W, (8) 52"" 41' 6"N & 107"" 58' 59"W, (9) 52"" 27' 32"N & 111"" 38' 6"W** and, **(10) 50"" 23' 17"N & 113"" 17' 31"W, (11) 51"" 7' 55"N & 99"" 32' 24"W, (12) 52"" 50' 42"N & 101"" 25' 23"W, (13) 54"" 1' 52"N & 101"" 5' 2"W, (14) 54"" 00' 43"N & 101"" 52' 30"W, (15) 53"" 26' 49"N & 103"" 27' 22"N, (16) 54"" 57' 11"N & 116"" 17' 38"W, (17) 58"" 18' 14"N & 115"" 41' 31"W, (18) 58"" 29' 31"N & 118"" 24' 11"W, (19) 57"" 44' 20"N & 117"" 14' 10"W, (20) 55"" 55' 55"N & 120"" 51' 00"W** and **(21) 52"" 48' 25"N & 115"" 52' 48"W**.

# Appendix III

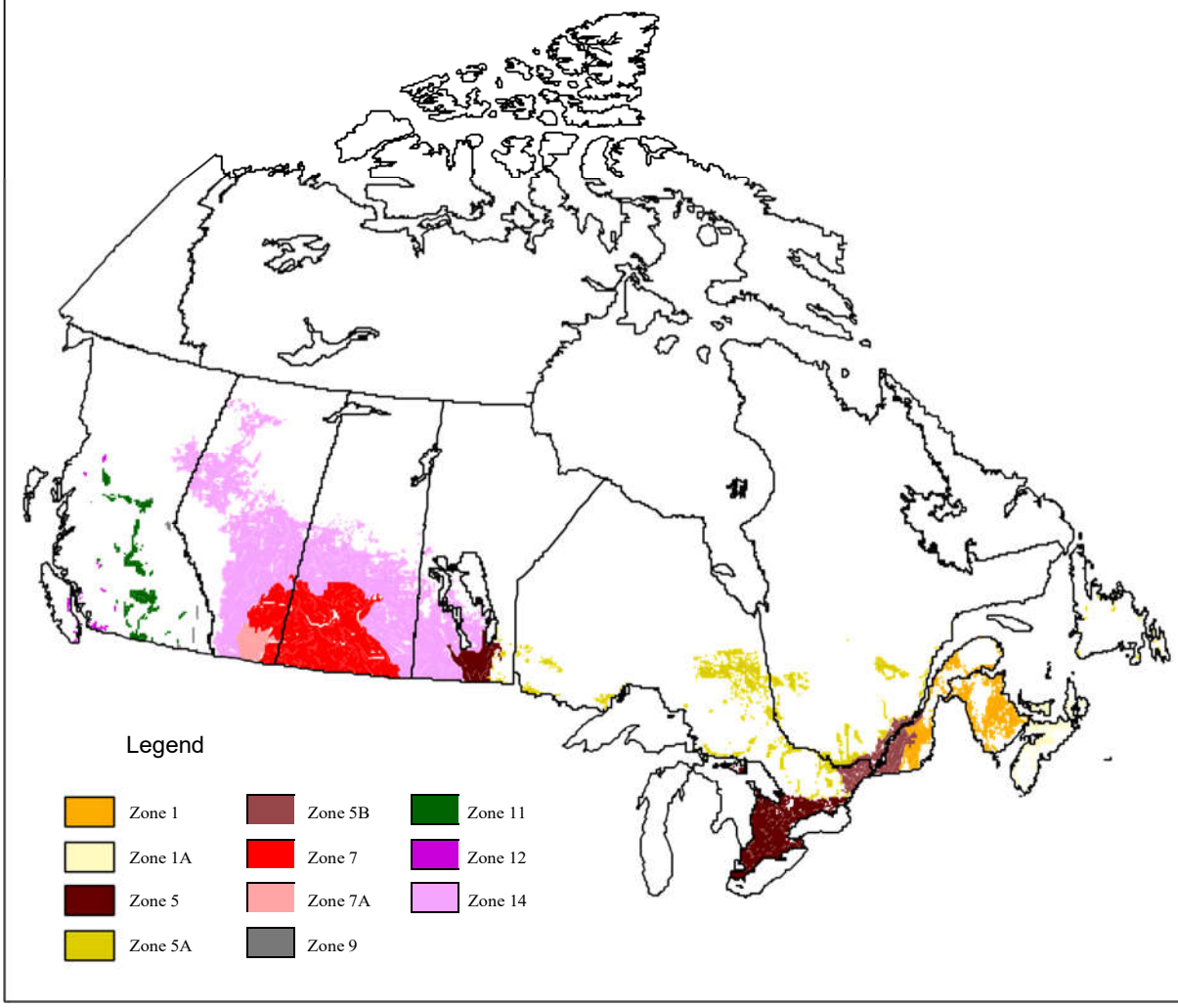
Maps defining Canadian major and minor crop field trial regions




Prepared for Pest Management Regulatory Agency, Health Canada

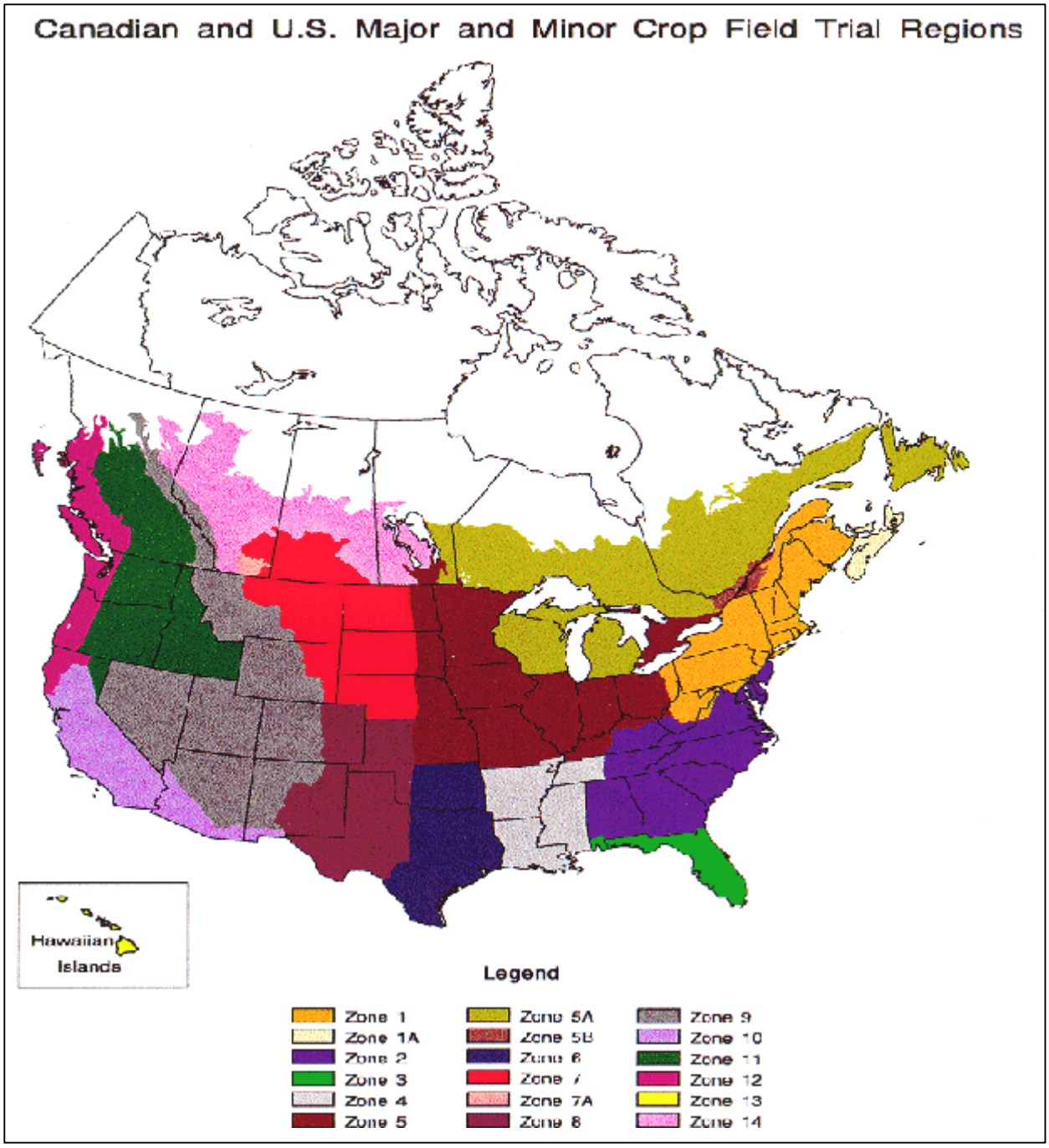
Produced by SAGA, Agriculture Division, Statistics Canada

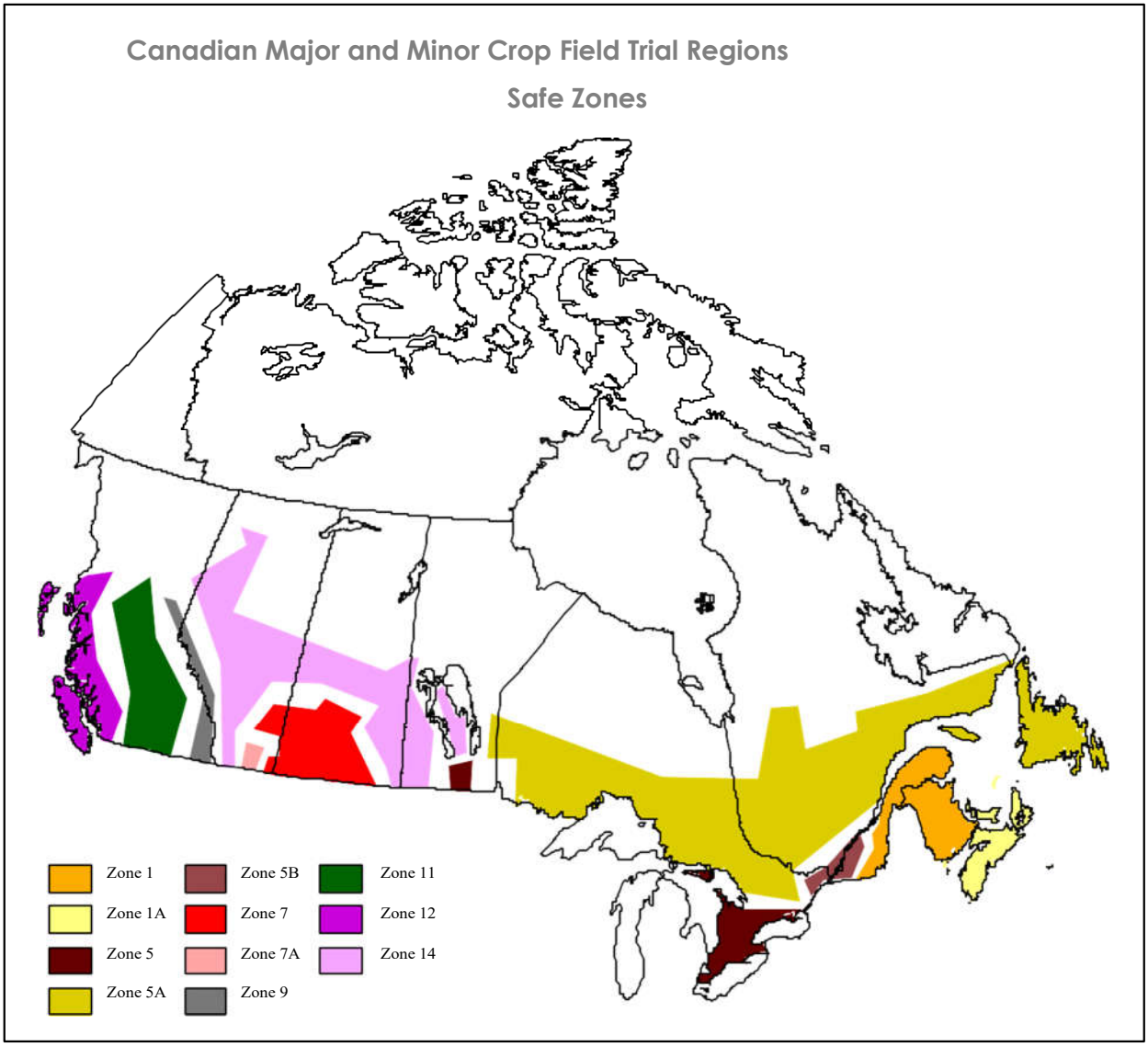
## Major and Minor Crop Field Trial Regions within Canadian Arable Lands




Prepared for Pest Management Regulatory Agency, Health Canada

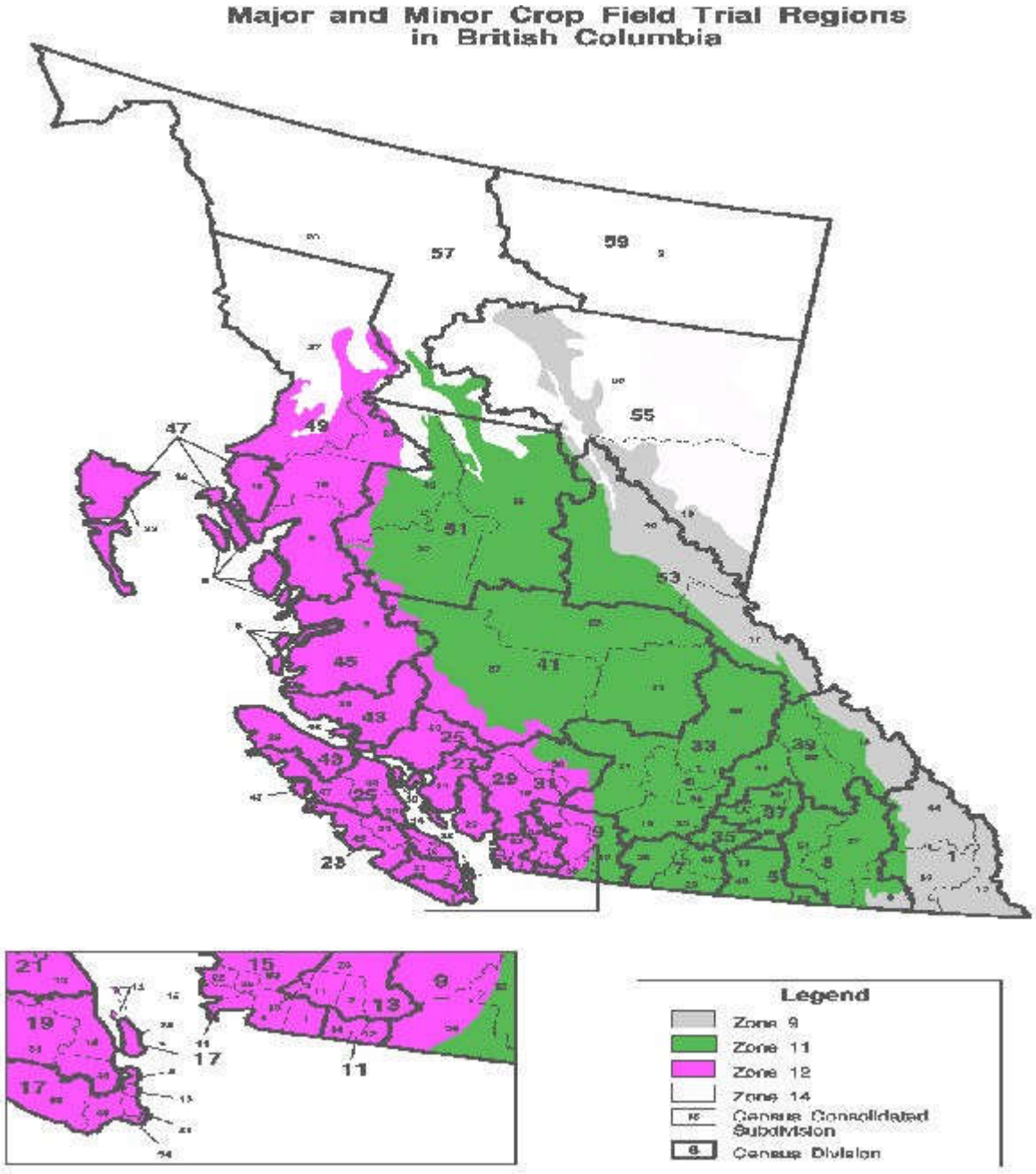
Produced by SAGA, Agriculture Division, Statistics Canada


Canadian and U.S. Major and Minor Crop Field Trial Regions
Hawaiian Islands
Legend
Zone 1
Zone 1A
Zone 2
Zone 3
Zone 4
Zone 5
Zone 5A
Zone 5B
Zone 6
Zone 7
Zone 7A
Zone 8
Zone 9
Zone 10
Zone 11
Zone 12
Zone 13
Zone 14

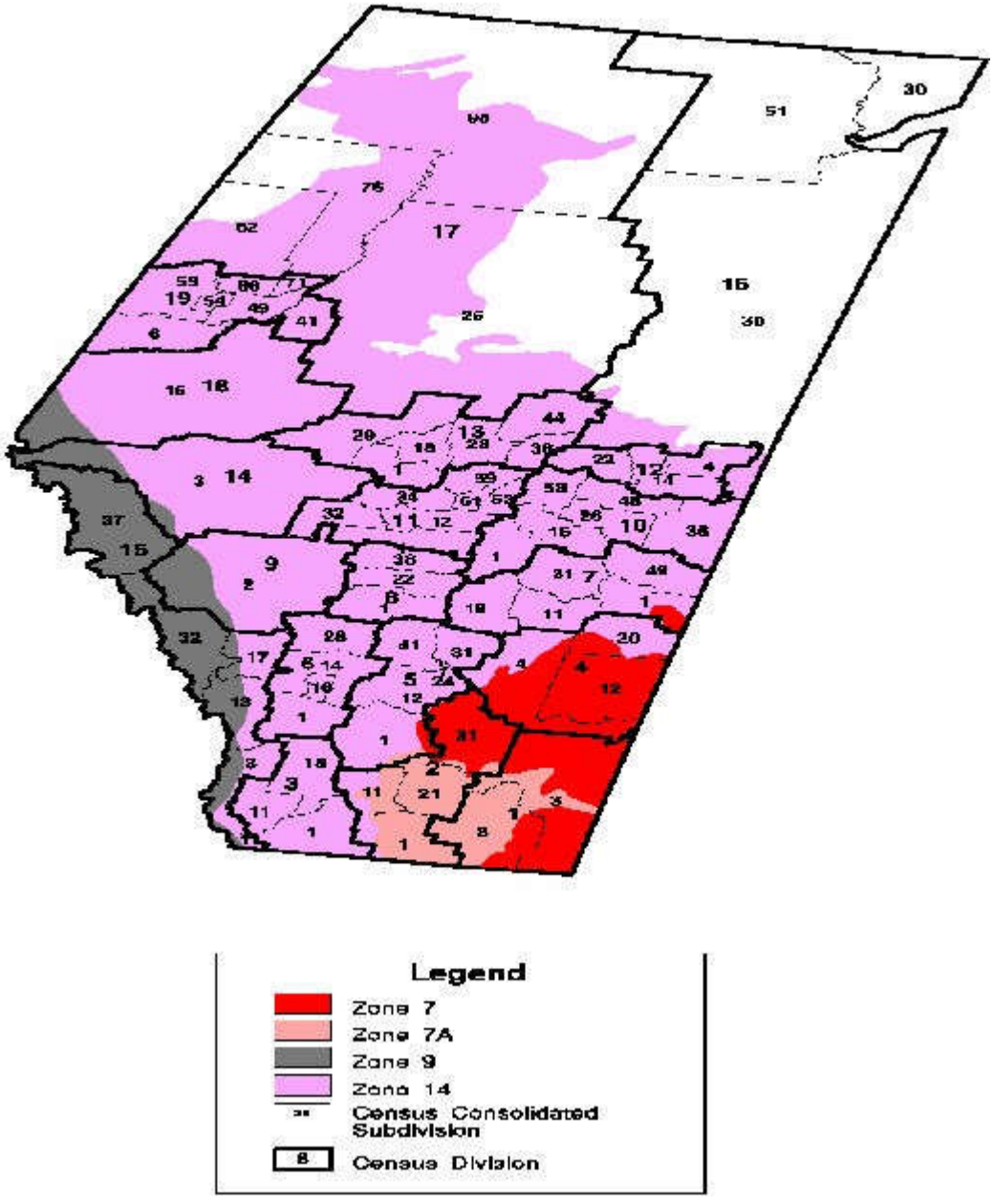



*Prepared for Pest Management Regulatory Agency, Health Canada*

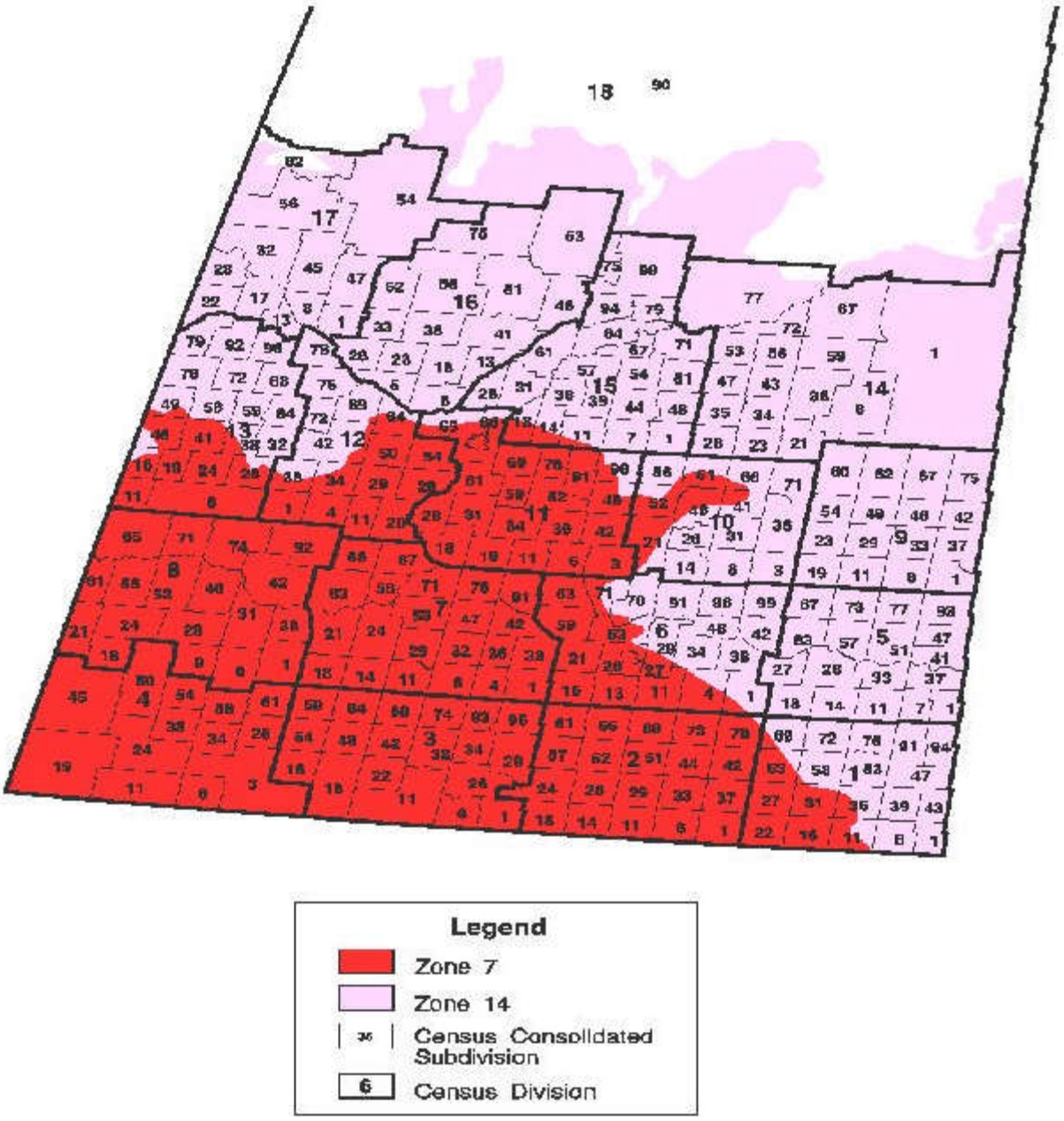
*Produced by SA GA, Agriculture Division, Statistics Canada*


Major and Minor Crop Field Trial Regions
in British Columbia
Legend
Zone 9
Zone 11
Zone 12
Zone 14
Census Consolidated Subdivision
Census Division

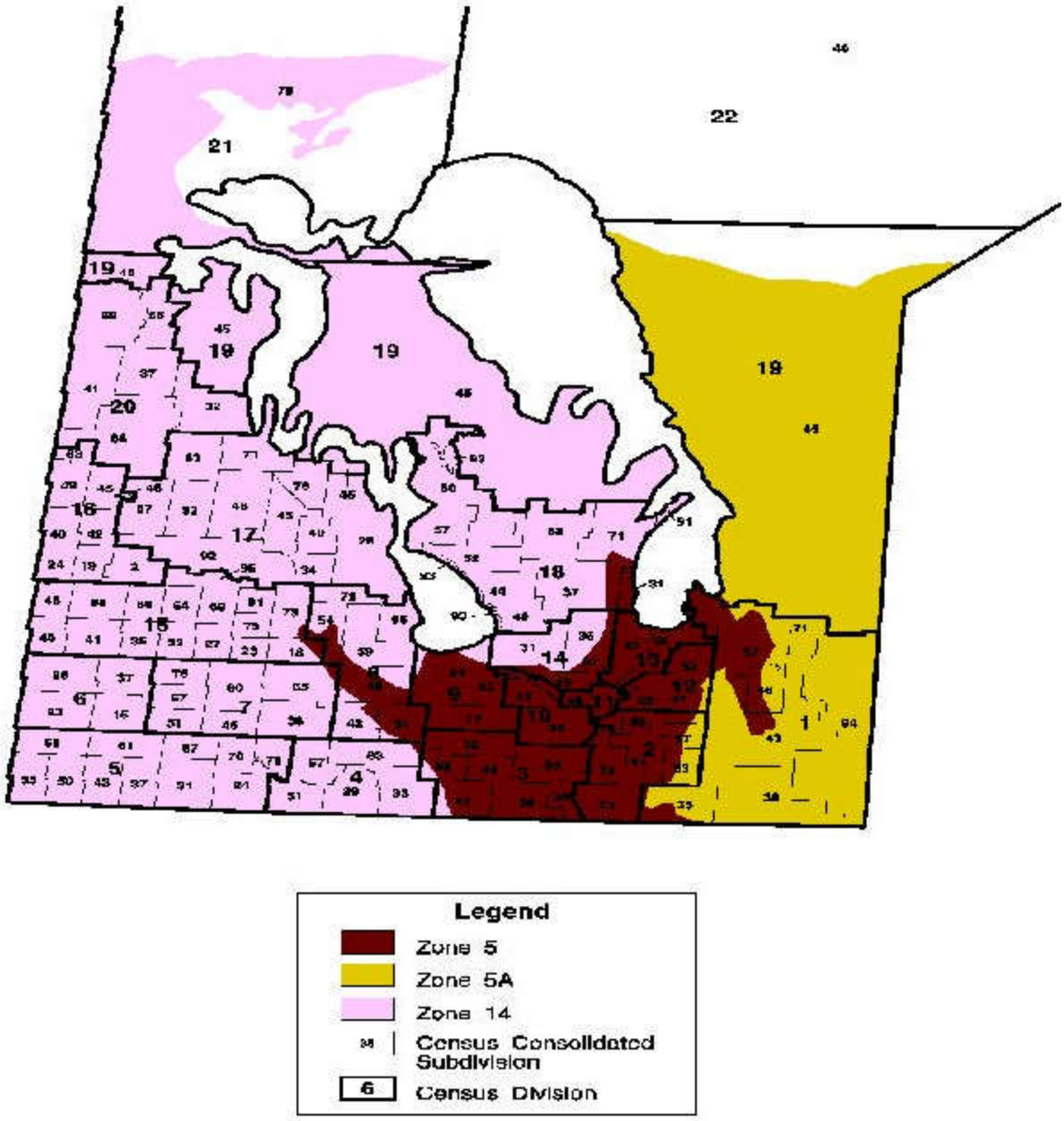
## Major and Minor Crop Field Trial Regions In Alberta



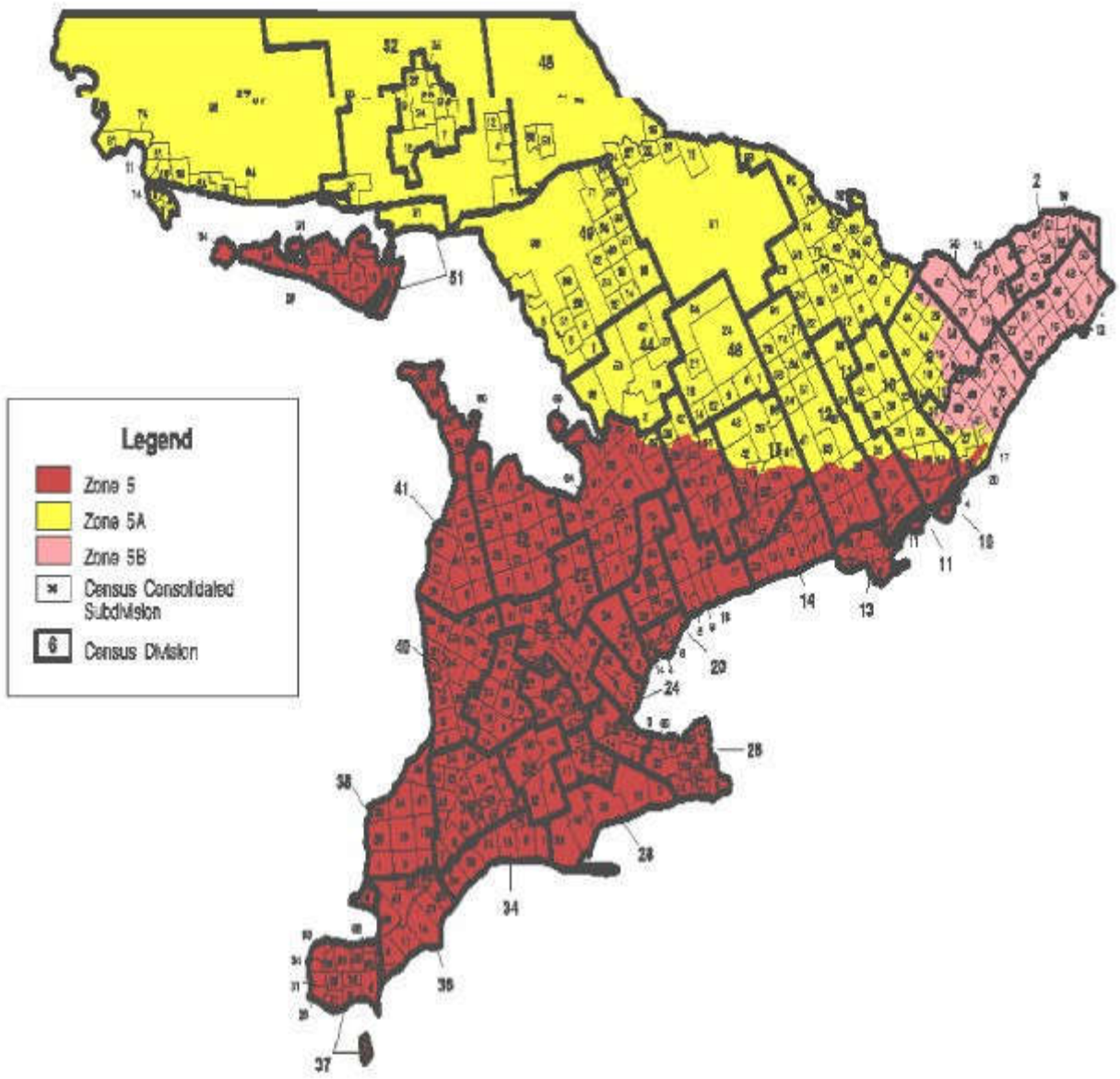
## Major and Minor Crop Field Trial Regions in Saskatchewan

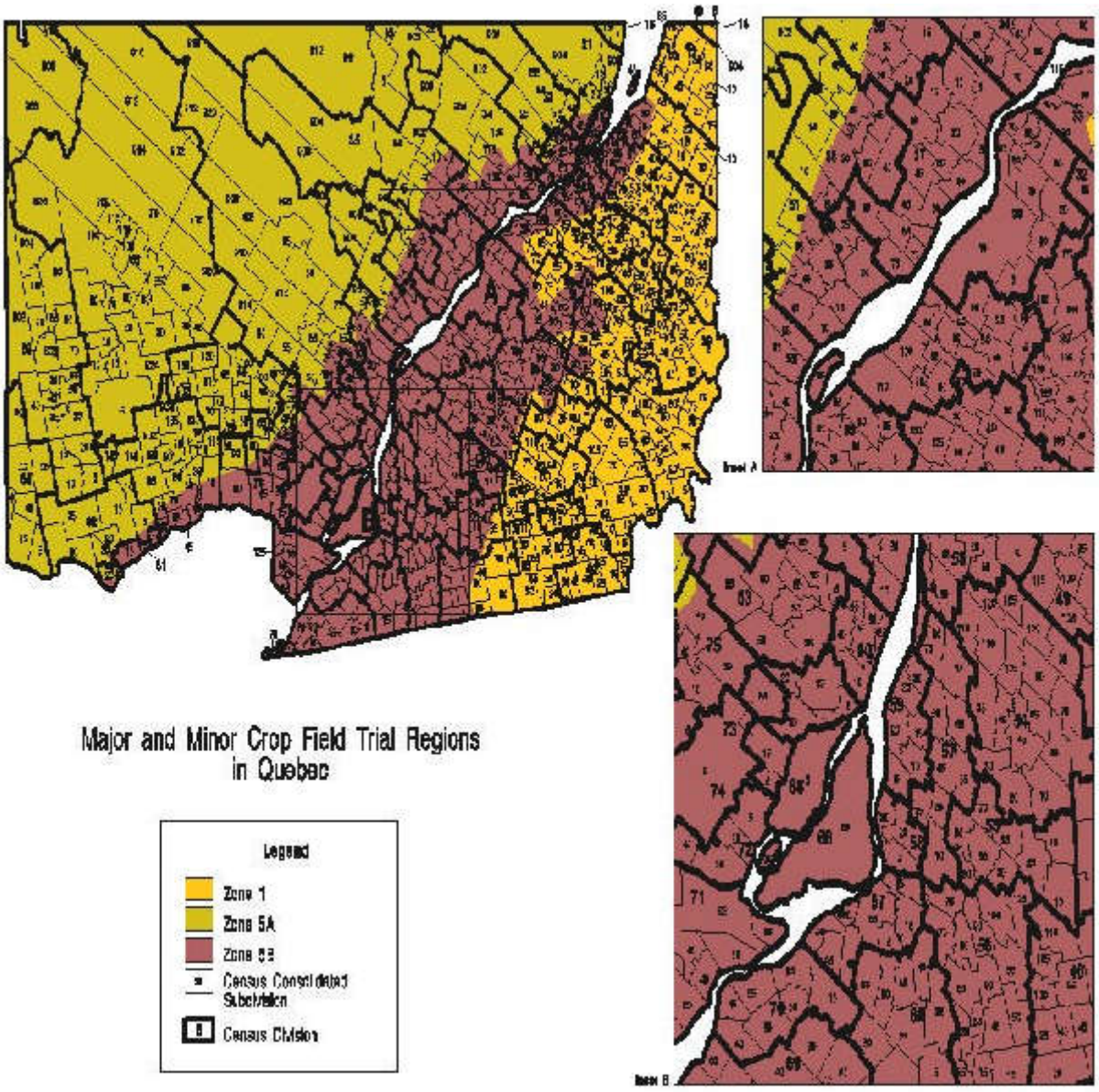


## Major and Minor Crop Field Trial Regions in Manitoba



Major and Minor Crop Field Trial Regions in Ontario






Major and Minor Crop Field Trial Regions in Quebec