B.7.6 Residues Resulting from Supervised Trials

 (Annex IIA 6.3; Annex IIIA 8.3)

B.7.6.1 Residues in Target Crops

**B.7.6.1.1 [Crop or Crop Group 1]**

**Document ID:** MRID No.

PMRA No.

Report: Report Citation

**Guidelines:** EPA OCSPP Harmonized Test Guideline 860.1500 Crop Field Trials (August 1996)
PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 9 – Crop Field Trials

PMRA Regulatory Directive DIR2010-05 – Revisions to the Residue Chemistry Crop Field Trial Requirements
OECD Guideline 509 Crop Field Trial (September 2009)

**GLP Compliance:** [No or Significant] deviations from regulatory requirements were reported which would have an impact on the validity of the study. [If “Significant,” then explain below the deficiencies and their impact on the acceptability of the study]

**Acceptability:** The study [is/is not] considered scientifically acceptable. [If not acceptable, then explain why below]

**Evaluator:** [Name of regulatory person who reviewed the study]

**EXECUTIVE SUMMARY**

[Number] field trials for [active ingredient] on [crop(s)] were conducted in Canada and/or the United States encompassing North American Free Trade Agreement (NAFTA) Growing Regions [List Regions and State or Province; # of trials] during the [year] growing season.

At each trial location, [describe timing and method of application; formulation used, rate, treatment interval and seasonal application rates of [xx] lbs ai/A (xx g ai/ha)]. An adjuvant [was or was not] added to the spray mixture for all applications. [Crops] were harvested at a preharvest interval (PHI) of [xx] days. In [one] trial, samples were collected at different time intervals (PHIs of x, xx, xxx days) to monitor residue decline.

All samples were maintained frozen at the testing facility, during shipping to the laboratory, and were stored frozen until analysis. The maximum storage interval for samples between harvest and analysis was [xx] days/months. Residues of [active ingredient] have been shown to be stable in [crops] for up to [xx] days under frozen conditions. Adequate storage stability data are therefore available to support the storage conditions and intervals for samples in the current trials.

Samples in the current study were analyzed using Method [Method ID], a [describe method] to determine residues of [list analytes]. Acceptable [method validation and] concurrent recoveries were reported for [matrices] samples at fortification levels of [xx] mg/kg (ppm), thus validating the method. The limit of quantitation (LOQ) was [xx] ppm per analyte for [matrices].

Individual sample (and per-trial average) residues in [matrix] ranged from [xx] ppm to [yy] ppm ([xx] ppm to [yy] ppm). [Include for each matrix and/or variation in use pattern in the study]. Residue decline data show that residues of [active ingredient] [increase/decrease/are unchanged/are too variable to assess decline] in [commodities] with increasing PHIs.

[Include this section only if the "GLP Compliance" prompt above is answered "Significant deviations from regulatory requirements were reported."]

**COMPLIANCE**

The following deviations from GLP requirements were reported: [list].

[Include this section only if the "Acceptability" prompt above is answered "The study is not considered scientifically acceptable."]

**STUDY DEFICIENCIES**

Under the conditions and parameters used in the study, the data are classified as scientifically unacceptable. [Explain the deficiencies and their impact on the acceptability of the study.] The study [can or cannot] be upgraded by submission of additional information; if “can be,” then list the additional data required.

**I. Materials and Methods**

**A. Materials**

|  |
| --- |
| **Table B.7.6.1.1-1. Nomenclature for [Active Ingredient] and Metabolites of Interest.** |
| **Common name** | (active ingredient) |
| **Identity** | [CAS Chemical Name] |
| **CAS no.** |  |
| **Company experimental name** |  |
| **Other synonyms (if applicable)** |  |
|  |
| **Metabolite X** | (for each analyte) |
| **Identity** | [CAS Chemical Name] |
| **CAS no.** |  |
| **Company experimental name** |  |
| **Other synonyms (if applicable)** |  |

**B. Study Design**

**1. Test Procedure**

A total of [xx] residue trials in/on [crop] were conducted with a [formulation] during the [year] growing season(s) (Table B.7.6.1.1-2).

|  |
| --- |
| **Table B.7.6.1.1-2. Trial Numbers and Geographical Locations.** |
| Crop | Region | Total |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Crop 1 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Crop 2 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Etc. |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

Locations and detailed use patterns for the trials are provided in Table B.7.6.1.1-3.

| **Table B.7.6.1.1-3. Study Use Pattern.** |
| --- |
| Location: City, State/Province; Year (Trial ID) | End-use Product/ Formulation (% ai) | Method of Application/ Timing of Application | Volume(gal/A)[L/ha] | Rate per Application(lbs ai/A)[g ai/ha] | Retreatment Interval (days) | Total Rate(lbs ai/A)[g ai/ha] | Surfactant/ Adjuvant |
|  |  | 1. |  |  |  |  |  |
| 2. |  |  |  |
| 3. |  |  |  |
|  |  | 1. |  |  |  |  |  |
| 2. |  |  |  |
| 3. |  |  |  |

[Note: If crop is cotton, then add a column for harvesting procedure.]

[Crops] were grown and maintained according to typical agricultural practices. Irrigation was/was not used. No unusual weather conditions were reported during the study [or report any anomalies].

**Sample Handling and Preparation**

[Briefly describe how samples were handled after harvesting (shipment, storage, etc.) and any preparation that was done prior to extraction.]

**2. Description of Analytical Procedures**

Samples of [crop] were analyzed for residues of [analyte(s)] using the Analytical Method [ID# and Title]. [Indicate if the method was previously reviewed and/or validated and for what commodities.]

[Reference study summary if method is described in the B.5.2 section of this review, or provide a description similar to that below if it is a different method.]

Briefly, samples were extracted with [solvent system]. Extracts were cleaned up using [SPE column, partitioning, etc.] and a portion of this extract was analyzed for residues of [list analytes] using [describe instrument/detector system]. The LOQ was [xx] ppm for each analyte. [State the LOD if available and how the LOQ and LOD were determined.]

**II. RESULTS AND DISCUSSION**

Method performance was evaluated [during method validation and] by use of concurrent recovery samples by fortifying [matrix] at [xx and yy] ppm. [n] samples of [crop matrix] were fortified at [xx] ppm and individual recoveries ranged from xx% to yy% with a standard deviation of [xx%]. [n] samples of [crop matrix] were fortified at [yy] ppm and individual recoveries ranged from xx% to yy% with a standard deviation of [xx%]. All recoveries were within the acceptable range of 70% to 120%; therefore, the method was considered valid for the analysis of [active ingredient and metabolites] residues in [crop] matrices (Table B.7.6.1.1-4). [Note Table B.7.6.1.1-4 should only be included if recoveries are consistently outside the acceptable range.] The fortification levels [did/did not] bracket the measured residues.

The detector response was linear (coefficient of determination, r2 >xx) within the range of [concentrations]. Representative chromatograms of control samples, fortified samples and treated samples were provided. The control chromatograms generally had no peaks of interest above the chromatographic background. [The fortified sample chromatograms contained only the analyte of interest, and peaks were symmetrical and well defined.] or [Residues in controls were ≤xx ppm. The reported residue values [were/were not] corrected for apparent residues in controls.] Metabolites were expressed in parent equivalents (if study did not, the reviewer may need to do so).

|  |
| --- |
| **Table B.7.6.1.1-4. Summary of Procedural/Concurrent Recoveries of [Active Ingredient] from [Matrix]1.** |
| Matrix | Fortification Level (ppm) | Recoveries(%) | Mean ± Std. Dev.(%) |
| [Analyte] |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

1 This table should be included only if recoveries are consistently outside the acceptable range.

The field residue samples were stored frozen a maximum of [xx days/months] from harvest to analysis (Table B.7.6.1.1-5). [Table B.7.6.1.1-5 should only be included if storage stability data are not included in B.7.6.2; if it is included elsewhere in the document, just cite location in monograph.]

The available freezer storage stability data indicate that residues of [active ingredient and metabolites (if applicable)] were stable when stored frozen at ≤-20oC in [crop(s)] for up to [demonstrated period]. [Indicate if the freezer storage stability data were previously reviewed and report the demonstrated storage intervals for each matrix/analyte]; or

Freezer storage stability data were generated concurrently with the [crop] field trials. [Note: A summary table of these results should be inserted here.] Data showed that [active ingredient and metabolites (if applicable)] residues were stable in [matrices] under frozen storage for the duration of the storage period.

|  |
| --- |
| **Table B.7.6.1.1-5. Summary of Storage Conditions1.** |
| Matrix(RAC or Extract) | Storage Temperature (°C) | Actual Storage Duration(days/months) | Interval of Demonstrated Storage Stability[specify crop/matrix if different](days/months) |
|  |  |  |  |
|  |  |  |  |

1 Delete this table if storage stability addressed in B.7.6.2.

The results from these trials showed that when harvested [xx] days after the last of [number] application(s) at a seasonal rate of [xx] lbs ai/A ([xx] g ai/ha), residues of [active ingredient and metabolites, if applicable] in [crop matrices] ranged from [xx] ppm to [yy] ppm (Tables B.7.6.1.1-6 and B.7.6.1.1-7).

[Select appropriate option from four choices: “Due to the very low levels of observed residues, no decline trend could be determined in [crop].”; or “In the residue decline trials, mean residue level decreased from [xx] ppm to [yy] ppm in [crop matrix] between PHIs of [xx] and [yy] days.”; or “Residues in [crop matrix] remained approximately the same through the [xx] days of residue decline.”; or “Residues in [crop matrix] were too variable to assess decline.”]

| **Table B.7.6.1.1-6. Residue Data from [Crop] Field Trials with [Active Ingredient].** |
| --- |
| Location: City, State/Province; Year (Trial ID) | Region | Crop/ Variety | Matrix | End-Use Product | Rate(lbs ai/A)[kg ai/ha] | PHI(days) | Residues1 (ppm) |
| Analyte 1 | Analyte 2 | Analyte 3 | Total2,3(per-trial average) |
| Trial 1 |  |  |  |  |  |  | Rep 1 | Rep 1 | Rep 1 |  |
| Rep 2 | Rep 2 | Rep 2 |
| Trial 2 |  |  |  |  |  |  | Rep 1 | Rep 1 | Rep 1 |  |
| Rep 2 | Rep 2 | Rep 2 |
| Trial 3 |  |  |  |  |  |  | Rep 1 | Rep 1 | Rep 1 |  |
| Rep 2 | Rep 2 | Rep 2 |
| Trial 4 |  |  |  |  |  |  | Rep 1 | Rep 1 | Rep 1 |  |
| Rep 2 | Rep 2 | Rep 2 |

1 Expressed as parent equivalents.

2 Total = Parent + Metabolite X [which corresponds to the residue definition for enforcement purposes].

3 Do not include this column if the residue definition for enforcement purposes has not been determined.

[Note: When the residue definition (RD) is different for tolerance/enforcement and risk assessment, the residues corresponding to the RD for risk assessment are reported in the dietary exposure assessment (risk assessment) template only.]

[DO NOT include the following table if the RDs are not determined at the time of the primary review. This table will be included only in the overview document (Level D Review) if the RDs are not determined. The statistics are compiled only for the RDs, not for each individual analyte.]

|  |
| --- |
| **Table B.7.6.1.1-7. Summary of Residues from [Crop] Field Trials with [Active Ingredient].** |
| Crop Matrix | Analyte | Total Application Rate (lbs ai/A) [g ai/ha] | PHI (days) | n | [Specify which residues, e.g. Combined Residues of A and B]Residues1 (ppm) |
| Max.2 | LAFT3 | HAFT3 | Median3 | Mean3 | SD3 |
| CROP 1  |
|  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |
| CROP 2 |
|  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |

1 Expressed as parent equivalents.

2 Values based on total number of samples.

3 Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (xx ppm).

n = number of field trials.

**III. CONCLUSIONS**

The [crop] field trials are considered scientifically [acceptable or unacceptable]. The results of the study showed that following a total application of [rate] in [crop] samples collected at PHIs of [xx] days, [active ingredient] residues ranged from [xx] ppm to [yy] ppm. A decline study indicates that the level of [active ingredient] residues in [crop] [increases/decreases/remains constant] with time. Adequate storage stability data are available to support sample storage durations and conditions. [Also, specify for metabolites, if applicable.]

**REFERENCES**

[Cite references for analytical methods and freezer storage stability studies. Include the EPA MRID# and PMRA# of both the study and the review (if available)].

**B.7.6.1.2 [Crop or Crop Group 2]**

**Document ID:** MRID No.

PMRA No.

**Report:** Report Citation

**Guidelines:** EPA OCSPP Harmonized Test Guideline 860.1500 Crop Field Trials (August 1996)
PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 9 – Crop Field Trials

PMRA Regulatory Directive DIR2010-05 – Revisions to the Residue Chemistry Crop Field Trial Requirements
OECD Guideline 509 Crop Field Trial (September 2009)

**GLP Compliance:** [No or Significant] deviations from regulatory requirements were reported which would have an impact on the validity of the study. [If “Significant,” then explain below the deficiencies and their impact on the acceptability of the study]

**Acceptability:** The study [is/is not] considered scientifically acceptable. [If not acceptable, then explain why below]

**Evaluator:** [Name of regulatory person who reviewed the study]

**[Repeat previous sections, modify as appropriate.]**

Template Version – February 2016