B.7.6 Residues Resulting from Supervised Trials

 (Annex IIA 6.3)

B.7.6.2 Stability of Residues Prior to Analysis

**B.7.6.2.1 [Crop(s)]**

**Document ID:** MRID No.

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**Report:** Report Citation

**Guidelines:** EPA OCSPP Harmonized Test Guideline 860.1380 Storage Stability Data (August 1996)
PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 5 – Storage Stability Data
OECD Guideline 506 Stability of Pesticide Residues in Stored Commodities (October 2007)

**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**

Samples of [ground or whole crop/matrix] were fortified with [analytes] at a level of [fortification level] and put into storage at [temperature]. At intervals of [xx, yy, and zz] months, stored samples and freshly fortified samples were analyzed for residues of [list analytes].

At each storage interval, [analytes] were determined using Method [Method ID], a [describe method]. 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].

Under these conditions, residues of [active ingredient and metabolites (if applicable)] were stable {or [decreased or increased] by [percentage]} in [crop/matrix] for [duration of time].

[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**

**1. Test Material**

|  |
| --- |
| **Table B.7.6.2.1-1. 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)** |  |

**2. Test Commodity**

**Matrix: [list matrices]**

**B. Study Design**

**1. Test Procedure**

[Briefly describe the fortification procedure, including the solvent used for the standard fortification solution, the concentration, the stability of this solution, the condition of the matrix at the time of fortification (e.g., extract, homogenate, macerate, etc.), the time allowed for equilibrium etc.]

For example…

Untreated samples of [crop matrix] were acquired from [specify location]. The samples were transferred individually from freezer storage and processed from frozen by homogenization with dry ice using a Robot Coupe blender. The samples were returned to freezer storage after processing and left to allow dissipation of the dry ice prior to use.

A total of [xx] sub-samples of each of the three different matrices were weighed into polyethylene bottles. For each matrix, [xx] sub-samples were used as control samples and procedural recovery samples. The remaining [xx] sub-samples were fortified with each analyte individually at [yy] ppm and were to be used for storage stability samples.

On the day of sample fortification (0 day), a single control, duplicate samples fortified with [yy] ppm of [active ingredient], duplicate samples fortified with [yy] ppm of [metabolite 1], and duplicate samples fortified with [yy] ppm of [metabolite 2] were analyzed. The remaining samples were transferred to freezer storage (-20°C) until required for analysis. Samples were analyzed after storage intervals of approximately [1, 3, 6, 12, and 18] months and reported that actual storage periods were 31 days (1 month), 92 days (3 months), 182 days (6 months), 365 days (12 months), and 542 days (18 months). At the specified storage interval, a set of samples for each crop matrix was removed from frozen storage for analysis. The sample set for each storage interval consisted of the same number of samples as the sample set from the day-0 analysis. No concurrent recovery samples were run for the day-0 sampling set.

**2. Description of Analytical Procedures**

Samples of [matrix] 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 fortification [matrix] at [xx] and [yy] ppm. 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.7.6.2.1-2). Concurrent recoveries were used to correct the residues in the stored samples.

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.] Metabolites were expressed in parent equivalents (if study did not, the reviewer may need to do so).

|  |
| --- |
| **Table B.7.6.2.1-2. Summary of Procedural/Concurrent Recoveries of [Active Ingredient] from [Matrix].** |
| Matrix | Fortification Level(ppm) | Recoveries(%) | Mean ± Std. Dev.(%) |
| [Analyte] |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

[Discuss the storage stability of the analyte(s) during the tested storage intervals. If there is noteworthy dissipation of the analytes, describe qualitatively and quantitatively (provide regression analysis if appropriate).]

|  |
| --- |
| **Table B.7.6.2.1-3. Stability of [Active Ingredient] Residues in [Matrices] During Storage at [xx]°C.** |
| Commodity | Storage Duration (days/months) | Fortification Level(ppm) | Recovered Residues(ppm) | Mean Recovery (%) | Concurrent Recovery (%) | Corrected Recovery1 (%) |
| [Analyte] |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |

1 Corrected for concurrent recovery.

[Insert a figure (Figure B.7.6.2.1-1) of % recovery over time if such a figure will facilitate describing the stability profile and any dissipation trends or modeling.]

**III. CONCLUSIONS**

The storage stability study with [matrices] is considered scientifically [acceptable or unacceptable]. The results of the study showed that residues of [analytes] are stable for at least [xx months] when stored at or below [yy°C]. [Describe any dissipation during storage and its impact on interpretation of residue studies.]

If storage stability was shown for the five crop commodity categories (high water, high oil, high protein, high starch, high acid), then discuss the finding that stability is assumed for all crops.

For example…

The corrected recoveries in the storage stability study suggest [active ingredient and metabolites] are stable in water-, starch-, protein-, oil-, and acid-containing materials for up to xx months when stored frozen.

**REFERENCES**

Cite references for analytical methods [include the EPA MRID# and PMRA# of both the study and the review (if available)].

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