Analytical method for [analyte(s)] in [soil, water, air, or other environmental medium]

|  |  |
| --- | --- |
| Reports: | ECM: [Provide full citation. Provide the MRID (first) if the review is unilateral.]  ILV: [Provide full citation. Provide the MRID (first) if the review is unilateral.] |
| Document No.: | [MRIDs xxxxxxxx & xxxxxxxx] |
| Guideline: | 850.6100 [U.S.]  8.2.2.1 [soil]; 8.2.2.2 [sediment]; 8.2.2.3 [water]; 8.2.2.4 [biota] [Canada]  [If this review is multilateral, also provide the guideline numbers under which participating agencies are reviewing the analytical method.] |
| Statements: | [Indicate whether the method validations were conducted in compliance with FIFRA GLP standards and whether signed and dated Data Confidentiality, GLP Compliance, Quality Assurance, and Authenticity Certification statements were provided for the method and ILV reports. If the validations were not conducted in compliance with FIFRA GLP standards, indicate why or how they deviated.] |
| Classification: | This analytical method is classified as [provide classification and very concise statement of any deficiencies that impacted the classification] [E.g.: *“… acceptable for (applicable residues). However, the independent laboratory validated limit of detection for (analyte X) is 10x higher than that stated in the ECM report.”*] [If multiple classification terminologies are needed for multilateral reviews, list or tabulate them.] |
| PC Code: | [xxxxxx] |
| Reviewer: | [Provide final reviewer(s)’s name Signature:  and title.] Date: [Type date of signature.] |

**Executive Summary**

[This section does not appear in the original T2S template. Table 1 is prepared in the on-line ECM Index[[1]](#footnote-1) format for ease of posting.]

This analytical method, [method ID], is designed for the quantitative determination of [analyte(s)] in [matrix/matrices] using [acronym of chromatograph/detector] (see Table 1). The method is quantitative for [the analytes or a subset thereof] at [the stated LOQ(s) of X µg/L or other value #x higher than the stated LOQ(s)]. The LOQ(s) [is/are] [less than/equal to/greater than] the lowest toxicological level of concern in [matrix/matrices]. [Briefly summarize any major issues discovered by the independent laboratory and state whether the method was modified to address them.]

**Table 1. Analytical Method Summary**

| Analyte(s) by Pesticide | MRID | | EPA Review | Matrix | Method Date | Registrant | Analysis | Limit of Quantitation (LOQ) |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Environmental Chemistry Method | Independent Laboratory Validation |
| [Pesticide &/or Degradate(s)] | [MRID] | [MRID] | [Leave blank] | [Water/Soil/ Sediment/Plant] | [##/##/##] | [Company Name] | [Acronym of column/ detector] | [#] [µg/L or µg/kg] |

1. **Principle of the Method**

[Briefly describe the analytical method (including any preparation, extraction, cleanup, analyte spiking, derivatization, and analysis steps) and the analytes that the method will quantify. Note whether, for any analytes, the independently validated limits of detection and quantification differ from those of the initial method validation.]

1. **Recovery Findings**

[Briefly indicate whether mean recoveries and relative standard deviations (RSD) were within guideline requirements (mean 70-120%; RSD ≤20%), *i.e.*, whether the method is quantitative, for each analyte and in each matrix.] [Repeat or expand Tables 2 and 3 for each matrix.]

Table 2. Initial Validation Method Recoveries for Analytes in [Matrix]

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Analyte | Fortification Level (units) | Number of Tests | Recovery Range (%) | Mean Recovery (%) | Standard Deviation (%) | Relative Standard Deviation (%) |
| [Analyte x] | [LOQ] | [7] | [#-#] | [#] | [#] | [#] |
| [10x LOQ] | [7] | [#-#] | [#] | [#] | [#] |

Table 3. Independent Validation Method Recoveries for Analytes in [Matrix]

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Analyte | Fortification Level (units) | Number of Tests | Recovery Range (%) | Mean Recovery (%) | Standard Deviation (%) | Relative Standard Deviation (%) |
| [Analyte x] | [LOQ] | [5] | [#-#] | [#] | [#] | [#] |
| [10x LOQ] | [5] | [#-#] | [#] | [#] | [#] |

1. **Method Characteristics**

[This section combines the “Linearity,” “Specificity,” Limit of Quantitation,” “Repeatability,” and “Reproducibility” sections in the original T2S template.]

[Briefly state how the LOD and LOQ were calculated and whether the calculation procedures are scientifically accepted.] [Provide in Table 4 the limits of quantitation (LOQ) and detection (LOD) established by the independent laboratory validation (ILV). For linearity, provide the correlation coefficient (r2) and concentration range for the calibration curve. The linearity is satisfactory when r2 ≥ 0.995.[[2]](#footnote-2) State “Yes” where the method is satisfactorily repeatable, reproducible, and specific and provide a short explanation where the method is not. Repeatability is satisfactory when mean recoveries are 70-120% and RSDs are ≤20%. Reproducibility is satisfactory when the independent validation confirms the LOQ(s) established by the initial validation. Specificity is satisfactory when the method includes confirmation of analyte identity and there are no known interferences from the matrix, reagents, solvents, or equipment.]

Table 4. Method Characteristics

|  |  |  |  |
| --- | --- | --- | --- |
|  | [Analyte x] | [Analyte y] | [Analyte z] |
| Limit of Quantitation (LOQ) | [# µg/L] | [# µg/L] | [# µg/L] |
| Limit of Detection (LOD) | [# µg/L] | [# µg/L] | [# µg/L] |
| Linearity (calibration curve r2 and concentration range) | r2 = [#]  [# – # µg/L] | r2 = [#]  [# – # µg/L] | r2 = [#]  [# – # µg/L] |
| Repeatable | [Yes/No] | [Yes/No] | [Yes/No] |
| Reproducible | [Yes/No] | [Yes/No] | [Yes/No] |
| Specific | [Yes/No] | [Yes/No] | [Yes/No] |

1. **Method Deficiencies and Reviewer’s Comments**

[This section is titled “Conclusions” in the original T2S template.]

[List any deficiencies with the analytical method, the laboratory validations, and their documentation. Note whether deficiencies are with the method procedure, the laboratory validations, the documentation, or the method recoveries, and whether they affect the review classification. Some examples of deficiencies are as follows. Use of laboratory equipment that is not commercially available is a deficiency with the method procedure that would not affect the classification. Analyzing only three replicates per concentration and failing to analyze analytes at the LOQ are two deficiencies with the laboratory validation that would affect the method classification. Failing to provide representative chromatographs and failing to explain how the LOD and LOQ were calculated are two deficiencies with the documentation that would affect the method classification. (ECMs with poor documentation may be classified unacceptable even though they are valid and useful in practice.) Mean recoveries outside the range of 70-120% and/or with relative standard deviations greater than 20% typically invalidate a method and would affect the method classification. An LOQ above toxicological levels of concern does not invalidate the method, but results in an unacceptable method classification. If a major issue was discovered by the independent laboratory, then state whether the method was modified to address it and whether a new internal validation was performed.]

[If the initial validation was performed by a governmental agency, a reference to the agency’s documentation of the ECM will serve as the ECM report. More specifically, if the applicant submits an ILV report and documentation of the agency’s ECM, the initial validation report for the ECM is not needed. If the initial validation was performed by a private entity, the current applicant needs to submit two reports of performance data, as usual, one for the initial or other internal validation and one for the ILV.]

[Examples of satisfactory method reports, method reports with major deficiencies, and method reports with minor deficiencies are listed below:]

[Satisfactory method reports provide clearly written procedures for sample preparation, extraction, cleanup, derivatization (if required), and analysis. The procedures may be contained in a stand-alone analytical method report with a detailed narrative, a detailed flow chart, or both. The analytical procedures include information on the sample preparation technique, type of instrument and analytical column(s) used, instrument setup and operating parameters, standard and reagent preparation and calibration procedures. Method performance is demonstrated by acceptable recovery data (*i.e.*, method repeatability). Chromatograms for one standard, one matrix blank, and matrix spike at the LOQ and 10 x LOQ spiking levels with response values (*i.e.*, area counts) are included, as well as a regression analysis that defines the slope, intercept, and standard error of the calibration curve. Required equipment and glassware are generally available. An ILV has been performed on the same version of the analytical method and produced acceptable performance data, as described above, which are separate from those of the initial performance data. A report on the ILV findings accompanies the registrant's method report. Analysts seeking to validate a satisfactory method should be able to produce reliable and satisfactory data with minimal interpretation or additional instructions.]

[Method reports may be categorized as having major deficiencies with respect to the analytical procedures and/or performance data. Examples of major deficiencies with a method include lack of detail in the description of the analytical techniques, requirement of equipment or techniques not generally available, use of old or outdated methodologies or obsolete equipment, and lack of repeatability at or above the LOQ. Major deficiencies with a method report include lack of any performance data at the LOQ and 10 x LOQ and lack of an ILV report. Validation of methods with major deficiencies may not be possible without additional information from the registrant.]

[Method reports categorized with minor deficiencies provide adequate procedures for the sample preparation, extraction, cleanup, derivatization (if required), and analysis, but may lack the level of detail provided by satisfactory method reports. Procedures for standard and/or extraction procedures may lack detail and require interpretation on the part of the analyst. Method reports without sufficient performance data (*i.e.*, insufficient number of spiked samples and/or the absence of spikes at the LOQ and 10 x LOQ), sample chromatograms, and/or precision data obtained from sample spikes at the LOQ and 10 x LOQ are considered to have minor deficiencies. Generally, method reports with minor deficiencies will require greater interpretation and professional experience in order to produce acceptable data.]

[For further reference, the following is a list of common deficiencies reported for ECM reports:

* Calibration curves and representative chromatograms/spectra for each analyte measured in each matrix at all spiking levels were not provided.
* Copies were not provided of the chromatograms/spectra for the standards that were used to quantify the analyte(s).
* Example calculations were not provided showing how the raw data were converted to a final concentration.
* A statement was not provided to confirm that the scientists who developed the original ECM differed from those who performed the ILV.
* The method report has conflicting information on the method in different sections of the document. For example, the method report may list different columns in different sections.
* A confirmatory method such as mass spectrometry was not provided to confirm the identity of the compound.]

1. **References** [List any references cited in the review.]

**Attachment 1: Chemical Names and Structures**

**[**Attach a table (*i.e.*, structure table) of the chemical names, SMILES strings, CAS numbers, and structures of the analytes or refer to this table if it exists in a separate, associated document. Do not include multiple versions of chemical names and SMILES strings. Sources of data need not be included. However, formatting the structure table in conformance with the guidance for tabulating transformation product data for EFED ROCKS memoranda is recommended (with the right four columns left blank). At a minimum, repeat the table below for the analytes.

For multilateral reviews, chemical names, SMILES strings, structures, and CAS numbers are captured elsewhere in the Monograph. Therefore these data are not attached to each method review within the Monograph. When the Monograph is split into individual reviews in EFED’s files, however, either reference the Monograph’s structure table as a separate, associated document or attach it to each individual review.]

[Sample structure table with the minimum information needed.]

|  |  |
| --- | --- |
| **[Common name [list other common names] [if the same common name is used in different studies for different compounds, provide in parentheses the MRID associated with the common name for this compound.]]** | |
|  |  |
| IUPAC Name: | [Provide one IUPAC name.] |
| CAS Name: | [Provide one CAS name.] |
| CAS Number: | [Provide if available.] |
| SMILES String: | [Provide one SMILES string.] |
|  | |
| [Paste structure here.] | |
|  | |

[Sample EFED ROCKS memorandum format for structure tables.]



1. The ECM Index is found at <http://www.epa.gov/pesticides/methods/ecmindex.htm> (accessed Nov. 7, 2012). [↑](#footnote-ref-1)
2. This criterion is consistent with Superfund analytical methods for inorganic analytes at <http://www.epa.gov/superfund/programs/clp/download/ism/ism1nfg.pdf> (accessed Nov. 7, 2012). [↑](#footnote-ref-2)