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Environmental Protection
Agency

Prevention, Pesticides
And Toxic Substances
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EPA

Pesticide Reregistration Rejection Rate Analysis Environmental Fate

Follow-up Guidance for:

Submission of Raw Data

Guidance on Submission of Raw Data

Raw data, in the sense used here, are not the detailed laboratory records required for a GLP audit. The "raw data" required for an EFGWB review is the uninterpreted factual information that formed the basis for the study report. Enough data should be provided so that the EFGWB reviewer is able to understand how the experiment was performed and reconstruct the development of the study results.

As pointed out in the Environmental Fate "Rejection Rate Analysis" document, the need for additional raw data does not normally result in the rejection of a study, and is usually corrected by the submission of the required data. It is generally most efficient to provide a well-organized presentation of all potentially necessary data, rather than respond to requests for data during the study review. The data requirements listed below may not apply to all reviewers or all studies, but should provide a general outline of the kind of information necessary in order to make a reliable data evaluation.

General

Data presenting results from soil and/or water physicochemical characterization.

Data supporting the nominal test substance application rate, with calculations, where applicable.

All dates (not just intervals) associated with the study: site preparation, test substance preparation, dosing or application, sampling, extraction, analysis, etc. Tabulation with calculated intervals would be helpful.

The registrant should ensure that description of any referenced SOP's or standard methods must be available to the Agency at the time of the review. If these SOP's have been previously submitted to the Agency, the registrant may reference the documents by MRID or Accession number.

Information on reference substance characterization (as required by GLP's).

Techniques used for preparation of aliquots or subsamples.

If automated data calculation methods are used, some discussion/examples of the calculation techniques, such as spreadsheet formulae, calculation parameters from a commercial system, etc.

The quantitative data which form the basis for calculating the reported concentrations of radioactivity in all samples and extracts. This includes, but is not limited to, counting data (in dpm), sample weights, counting efficiencies, and other appropriate measurements so that concentration of

radioactivity may be verified. Data should be provided for control and treated samples. Background data for solvents should be included as well.

Supporting chromatograms, mass spectra, NMR spectra, IR spectra, UV-Vis spectra, and any other data used to characterize or identify degradation products. This would include chromatograms or spectra which demonstrate the absence of any proposed metabolites.

Quantitative data associated with chromatograms, autoradiograms, spectra, etc., so quantitative assessment of metabolite identification can be confirmed. This would include the amount of radioactivity (in dpm) used in the analysis and the amount recovered. A complete set of sample calculations should be included with formulae and variables defined in generally understood terms, using data from the submitted report.

Chromatograms of standards and controls.

Information on sample storage including conditions, sample form (extract or homogenized) and sample container. Often a range of temperatures is provided, which account for temperature spikes; information should also be included about the condition of samples during temperature spikes. Reports should detail how samples are handled and stored prior to receipt by the analyzing lab. In particular, the time between taking the sample and freezing the sample should be reported.

Analytical methods

Schematic diagram(s) of the analytical approach for the separation, identification, and quantitation of the test substance and its degradates.

Supporting validation data associated with all methods submitted for each matrix (soil, water, air, biological tissue, etc.). This would include not only final results but also sample weights, extraction volumes, final volume of extracts, peak heights/areas, injection volumes, and any other data which would allow the reviewer to reproduce the results. These data can be summarized in a table, and may be reported along with analytical data for treated samples.

Chromatograms for each matrix of interest at all spiking levels, including the claimed limit of quantitation. A minimum of ten chromatograms is suggested for each matrix including both spiked and control samples, particularly in those instances where tolerances are proposed at or near the limit of quantitation. There is a need for control and method blank chromatograms so reviewers can assess the

reported limits of detection and quantitation.

Chromatograms should not be merely "representative individuals," but should represent the range of all samples. Reviewers need to be able to assess reproducibility of the method, potential interferences, variations in signal-to-noise ratios, etc. If a study consists of less than 10 samples, than chromatograms from all samples should be provided. If there are more than 10 samples, at least 10 chromatograms should be provided.

A minimum of three chromatograms each of control and fortified samples in order to assess the limit of quantitation.

A sample calculation should be presented where a treated sample and fortified control are taken through the entire calculation procedure. A reviewer should be able to tie this calculation to a chromatogram and quantitative data included in the report.

Sufficient quantitative data should be provided so that a reviewer can independently calculate the results. This would include, but not necessarily be limited to, sample weights, extraction volumes, aliquot volumes/weights, injection volumes, final extract volumes, and peak heights/areas. Dates of each step should be specified so the treated sample data can be associated with control samples, spike samples, and standards. The data can be summarized in a spreadsheet.

The data for each calibration standard should be presented in a manner that allows the reviewer to calculate residues in/on treated samples, reproduce standard calibration curves, etc. from the <u>submitted data alone.</u>

Quantitative data detailing the recovery from spike samples. This is particularly important if sample recoveries are corrected for spike recoveries. It must be stated explicitly whether results are corrected for an average method recovery or the method recovery of the particular data set. If corrected values are reported for treated samples, then apparent values should be reported as well.

Notes on any technical communications between the study sponsor and the independent laboratory regarding method validity and validation. This could be summarized by the analyst in a report rather than being notes from the laboratory notebook.

Analyst notes on the difficulties of the method and modifications to facilitate method implementation.

All <u>appropriate</u> manufacturer and lot numbers for chromatography columns, chemicals, and equipment.

Field data

Field notes and/or reports on application, harvest, plot preparation and maintenance.

Procedure and results for calibration of application equipment.

A specific description as to how and where (within the field plot) samples were taken. What was done to insure that the sample was representative?

Daily wind, rain, and temperature data is required to determine the relationship between environmental conditions and pesticide dissipation.

Irrigation data, including date, quantity, and application method is required.

Soil series of test plots, with SCS description of a typical profile for that soil type.

Description of terrain, including percentage and direction of slope, and depth to the water table.

Additional considerations

No data should be included from analyses aborted due to equipment failure or other circumstances. The registrant is required to keep such data in accordance with GLP standards, but such data should not be submitted with the final study report.

Information should be provided in a manner which is complete and legible. It should be organized so that the reviewer can easily find the desired information, and not have to look in many different places in a report to find the dates or analytical results.