

PROTOCOL FOR  
RAW AGRICULTURAL COMMODITY (RAC) RESIDUE  
EVALUATION OF <TEST SUBSTANCE> APPLIED TO  
<TEST SYSTEM>

EPA Guideline Requirement

OPPTS 860.1500

Protocol Number



Prepared By

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**RAW AGRICULTURAL COMMODITY (RAC) RESIDUE EVALUATION OF  
<TEST SUBSTANCE> APPLIED TO <TEST SYSTEM>**

**SPONSOR:**

<Name>  
<Address>  
<Address>

**TESTING FACILITY:**

LANDIS INTERNATIONAL, INC.  
3185 Madison Highway  
Valdosta, GA 31603-5126  
Phone 229-247-6472

**STUDY DIRECTOR:**

<Name>  
LANDIS INTERNATIONAL, INC.  
3185 Madison Highway  
Valdosta, GA 31603-5126

**ANALYTICAL TESTING FACILITY:**

<Name>  
<Address>

**STUDY MONITOR**

<Name>  
<Address>

**PROTOCOL NUMBER:** <Protocol No.>

Trial No.	Location (EPA region)	Field Cooperator

**TEST SYSTEM:** <Test system>

**TEST SUBSTANCE:**

<Pesticide Trade Name>  
**Formulation:** <Percent active ingredient, type>  
**Lot No.:** Will be recorded in the study file  
**CAS No.:** <Number>  
**CAS Name:** <Name>  
**Common Name:** <Name>  
**EPA Reg. No:** <Number>  
**IUPAC Name:** <Name>

**RAW AGRICULTURAL COMMODITY (RAC) RESIDUE EVALUATION OF  
<TEST SUBSTANCE> APPLIED TO <TEST SYSTEM> (CONT'D)**

**PROTOCOL RECIPIENTS:**

- |                            |                           |
|----------------------------|---------------------------|
| (1) Sponsor                | (4) Field Cooperator      |
| (2) Study Director         | (5) Analytical Laboratory |
| (3) Quality Assurance Unit | (6) Study Monitor         |

**GLP NOTEBOOK RECIPIENT:**

- (1) Field Cooperator

**SAMPLE ACTIVITY I.D. LOG RECIPIENTS:**

- |                    |                           |
|--------------------|---------------------------|
| (1) Sponsor        | (3) Field Cooperator      |
| (2) Study Director | (4) Analytical Laboratory |

**PROPOSED EXPERIMENTAL START DATE:**

*<Expected date of application at  
earliest test site>*

**PROPOSED EXPERIMENTAL COMPLETION DATE:**

*<Date>*

## OBJECTIVE

The objective of this study is to provide data from the analysis of residues of *<test substance>* on raw agricultural commodity (RAC) samples of *<test system>* following application of this product at the maximum label rate. *<Test substance>* will be applied in *<number>* applications to each of the crops at *<amount>* lb ai/*<amount>* gallons to obtain the maximum labeled per season rate of *<amount>* lb ai/A. For all trials, *<test system>* will be treated with spray volumes of *<number>* GPA.

This study is designed to fulfill the requirements of OPPTS Guideline 860.1500. This study will be conducted in accordance with EPA, FIFRA, Good Laboratory Practice Standards (GLP); 40 CFR, Part 160 (October 1989).

## JUSTIFICATION OF TEST SYSTEM

**Application:** Various *<test system>* crops will be treated with *<test substance>* for control of *<target pest>*. A maximum seasonal application rate of *<amount>* lb ai/A will be applied by using *<number>* separate ground applications (*<amount>* lb ai/A in each application) at a *<number>* day interval on *<test system>* at *<number>* trial locations. This is the maximum labeled rate. The test substance is a typical end-use product and application and agronomic practices accurately reflect the proposed label and normal crop culture in the areas where the study will be conducted. All crops involved in this study will be destroyed at trial completion.

**Sampling System:** The sampling system described herein is the accepted convention for measuring the amount of pesticide residues on the raw agricultural commodity.

## IDENTIFICATION OF THE TEST SYSTEM

**Experimental Design:** The study will take place at *<number>* sites in *<state(s)>*. Each site will consist of one non-treated plot and one treated plot established where no *<test substance>* (or pesticide known to interfere with *<test substance>* or its metabolites) has been applied for at least 5 years. The plots will be designed using the guidelines in LANDIS INTERNATIONAL SOP #3.2-Current Revision, *Plot Construction and Identification* (see GLP Field Notebook provided by LANDIS INTERNATIONAL, INC.).

**Plot Size:** Each treated and non-treated plot will be composed of at least three *<dimension>* rows of *<test system>*. The non-treated plot will be minimum distance of *<distance>* ft upslope and upwind (slope direction supersedes wind direction) from the treated plot. The test plots may be designated areas within commercial fields. If a commercial field is used, the Field Scientist must have control over the maintenance pesticides used in the plots.

The plot size and design will be sufficient to obtain more than the required amount of crop material shown in the section labeled **Number of Residue Samples**, page 7.

## APPLICATION

**Test Substance:** The date of test substance shipment, lot number, date of receipt, and method of shipment, as well as the amount and container size, will be documented. The test substance will be stored in an appropriate manner and storage conditions and daily temperature extremes will be recorded. Subsequent to the final application of the test substance, the remaining *<test substance>* will be returned to LANDIS INTERNATIONAL, INC.

The Sponsor will obtain a purity analysis and characterization for each lot number of the test substance used. Information regarding the stability and homogeneity of the test substance will be part of the study raw data package. A retention sample from each batch of the test substance will be archived. All unused test substance and partially empty containers will be retained until the final report is signed, unless a prior waiver has been obtained from EPA. Test substance remaining at the completion of the study will be returned to *<Sponsor>*.

**Field Preparation:** The treated plot and control area should be managed according to agricultural practice in the area. Fertilization should be made according to normal agricultural practice and documented as to when and how applied, including rate and composition. Soil preparation and other cultural practices must be noted.

**Crop and Planting:** The crop variety and date of planting must be recorded. The variety should be one commonly grown in the area.

**Irrigation:** Irrigation will be applied as needed to assure good plant growth; however, no overhead irrigation that will dislodge test material residues should occur within six hours following an application. The dates of each irrigation event will be recorded in the field logbook.

**Maintenance Pesticide Applications:** All maintenance pesticide applications should be approved in advance by the Study Director whenever possible and always documented. Pesticides that do not interfere with *<test substance>* analysis may be applied in order to control weeds, pathogens, and other pests if they appear in the treated plot and/or control area. The treated plot and control area should be maintained with the same compound, rate, and at the same time.

**Pesticide Use History:** Provide an accurate and complete history of pesticide use for the test site. Include material applied, rate, and date of application, for three years prior to study initiation, as well as during the study.

**APPLICATION (CONT'D)**

**Application:** The crop will be treated using the following application parameters:

Test substance:	<Name>
Formulation:	<%a.i./type>
Carrier:	water
Rate:	<Active ingredient/area or volume>(± 10%)
Spray volume:	<Minimum labeled spray volume>
Number of applications:	<Number>
Timing:	<Spray interval(s)>
Application equipment:	<Type of sprayer/nozzle type>

These conditions were selected as those most likely to give representative residues, provide full coverage to the leaves, and to satisfy the EPA guidelines.

The application equipment will be calibrated according to appropriate SOPs prior to treatment. Calibration results will be sent to the Study Director after each application. Complete documentation will be recorded in the field logbook.

The actual application rate will be calculated based on output, the active ingredient concentration, and the application time or land area covered. Once the plot has been treated, the amount of product or spray volume will be checked and recorded as verification of the application rate. The remaining product material will be disposed of appropriately and in accordance with federal and state regulations.

There will be no other chemicals added to the tank mix except <test substance> and water unless noted in the study notebook. The source of the water used will be documented in the study record.

Applications must be made within <number> hour of mixing and applied at a time with little or no wind. The applications should be made when leaves are dry.

After each application, all data used for calculating rate and volume must be submitted to the Study Director in a timely fashion.

**Product and Water Samples:** For each field trial, <number> samples (approximately <amount> ml each) of test material, two-1 L samples of carrier water and three samples (approximately 15 ml each) of spray mix from the spray tank will be collected into appropriate containers at the time of each application. Test material and carrier water samples will be sent (ambient) to the analytical laboratory along with a photocopy of the calibration records from the GLP Field Notebook and a photocopy of the Application Verification Product/Water Sample Form via approved courier. Spray mix samples will be maintained frozen by the Field Cooperator. The Study Director may or may not elect to have these samples analyzed.

### SAMPLING

All samples will be taken in accordance with LANDIS INTERNATIONAL SOP #4.2-Current Revision, *Procedures to be Followed When Taking Samples of Plant Parts and Fruiting Structures* (see GLP Field Notebook provided by LANDIS INTERNATIONAL, INC.).

Raw agricultural commodity (RAC) samples will be collected <Number> days following the final treatment. The non-treated plot will be sampled before the treated plot and the non-treated samples will be handled and stored separately from treated samples. Each sample will consist of tissue taken from at least 12 indiscriminately selected plants in the subject plot. Each sample will be collected and composited from a separate pass through the plot. From the non-treated plot, one sample may be used for method development/validation and/or storage stability and one will be used for residue analysis. Both treated samples will be used for residue analysis and separately analyzed. The weight of each sample will be recorded in the GLP Field Notebook.

#### Number of Residue Samples: <Commodity>

PLOT	SAMPLE	MATRIX	MINIMUM WEIGHT (kg)*
Non-Treated	1 <sup>st</sup>	<Commodity>	<#>
Non-Treated	2 <sup>nd</sup>	<Commodity>	<#>
Non-Treated	Duplicate	<Commodity>	<#>
Treated	1 <sup>st</sup>	<Commodity>	<#>
Treated	2 <sup>nd</sup>	<Commodity>	<#>
Treated	1 <sup>st</sup> Duplicate	<Commodity>	<#>
Treated	2 <sup>nd</sup> Duplicate	<Commodity>	<#>

\* Collected from a minimum of 12 plants.

Special care must be taken to prevent contamination of the samples. After collection, RAC samples will be placed in appropriately labeled sample bags and stored in containers containing a coolant. The samples will be transferred to freezers as soon as possible and will be maintained frozen until analysis.

## SAMPLE HANDLING

All residue samples will be collected and frozen in residue bags provided by LANDIS INTERNATIONAL, INC. All samples will be packed and shipped according to LANDIS INTERNATIONAL SOP #4.14-Current Revision, *Packing and Shipping Samples*.

Packing/shipping forms provided by LANDIS INTERNATIONAL, INC., will be completed and the appropriate documents included for shipment (see LANDIS INTERNATIONAL SOP #5.12-Current Revision, *Cooperator and Laboratory Instructions for Sample Packing/Shipping Forms*).

The "**white**" and "**yellow**" copies of the packing/shipping form will be sent with the samples to the selected laboratory. Upon receipt at the laboratory, they will complete the section at the bottom of the form and retain the "**yellow**" copy for their files and return the "**white**" copy to LANDIS INTERNATIONAL, INC. The Field Study Scientist will provide the "**pink**" copy for LANDIS INTERNATIONAL, INC. archives and retain the "**gold**" copy for the GLP Field Notebook.

The samples will be shipped frozen via overnight or other approved courier to the following analytical laboratory:

### ANALYTICAL LABORATORY

<Name>

<Address>

On the day of shipping, the Field Study Scientist (Cooperator) must contact LANDIS personnel by TELEFAX (229 242-1562), by e-mail (projectmanager@landisintl.com) or by TELEPHONE (800 526-3471), with the following information:

- (1) Protocol and Trial Number
- (2) Test Chemical
- (3) Crop Treated
- (4) Shipment Destination
- (5) Courier and Airbill Number
- (6) Date Shipped and Expected Arrival Date
- (7) Sample Physical State: Frozen or Fresh
- (8) Sample Numbers or "SAIL" Line Numbers

## SAMPLE ANALYSIS

The analytical portion of the protocol will be added at a later date via protocol amendment.

## STATISTICAL ANALYSIS

Mean values will be calculated for summary purposes.



**RECORD KEEPING**

Original documents or legible verified copies of the following information must be furnished to the Study Director:

- (1) A description of the test substance (including lot number). The date of the test substance shipment, receipt and method of shipment as well as the amount and condition upon receipt will be documented. The dates and methods of disposal will be recorded.
- (2) A description of the test site, including a map of the test plots indicating their location in relation to a fixed point of reference, topography, size, location of the untreated plot in relation to the treated plot and a map of the general area (e.g., a county map) showing the test site in relation to the nearest town or city.
- (3) Cultural practices during the course of the trial. A detailed description of these practices will be included in the logbook.
- (4) Crop characteristics such as planting date, variety, stage of growth, size at application, and plant spacing.
- (5) The date and method of each application including calibration information.
- (6) Crop and pesticide use history for the trial area (treated plot and control area) for at least three years preceding the study.
- (7) Record of use and description of any maintenance fertilizers and pesticides.
- (8) A description of the type of irrigation, dates and amount applied.
- (9) Daily weather data from the nearest weather station for the length of the study, including air temperature, relative humidity, and wind speed and direction at application. Rainfall data must be collected from the test site. State the distance of the test site from the source of the weather data.
- (10) The date of each sampling and description of the sampling technique.
- (11) A record of sample handling, including time from collection to freezer and a copy of the freezer temperature log for the period of time the samples were stored.
- (12) A copy of the chemical storage temperature log for the critical period of time the test substance was stored (i.e., from receipt through the date of final application).
- (13) Record of test substance receipt, distribution, use at each trial site (test substance use log) and disposal.
- (14) All correspondence and other miscellaneous raw data needed to reconstruct the trial.
- (15) Documentation of crop destruction.

## REPORTING

A draft report will be issued to the Sponsor upon receipt of all pertinent information from the Field Study Scientist. Upon approval by the Sponsor, a final report will be issued. The report will include, but not be limited to the following:

- (1) Name and address of the facility performing the study and the dates on which the study was initiated and completed.
- (2) Objectives and procedures stated in the approved protocol, including changes in the original protocol.
- (3) The test and control articles identified by name, code number, composition, and stability, as supplied by the Sponsor.
- (4) A description of the analytical methods used.
- (5) A description of the test system.
- (6) A description of the application equipment and application procedures.
- (7) A description of all circumstances that may have affected the integrity of the study.
- (8) The name of the Study Director and other scientists and supervisory personnel involved in the study.
- (9) A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data, and a statement of the conclusions drawn from the analysis.
- (10) The signed and dated reports of each of the individual scientists involved in the study.
- (11) The locations where all samples, raw data, and the final report will be stored.
- (12) The statement prepared and signed by the Quality Assurance Unit which specifies the dates inspections were made and the dates findings were reported.
- (13) The signature of the Study Director on the final report.

Additions or corrections to the final report will be in the form of an amendment by the Study Director. The amendment will clearly identify that part of the final report that is being amended or corrected, the reasons for the additions or corrections, and will be signed and dated by the person responsible.

**QUALITY ASSURANCE**

LANDIS INTERNATIONAL will be responsible for assuring that the study practices conform to the U. S. Environmental Protection Agency Good Laboratory Practices (GLP). This protocol will be reviewed by the Quality Assurance Unit of LANDIS INTERNATIONAL and the Sponsor. The study will be audited at critical phases and reports of findings will be submitted to the Study Director and Study Director's Management. The study report prepared by LANDIS INTERNATIONAL will be examined by the Quality Assurance Unit of LANDIS INTERNATIONAL and the Sponsor and the review will be documented by the Quality Assurance Officer's signature on the final field report.

The Quality Assurance Unit of the analytical laboratory will provide the Study Director with all facility, data, process, and report audits to meet Environmental Protection Agency GLP requirements.

**PROTOCOL SIGNATURE PAGE**

\_\_\_\_\_

Sponsor

\_\_\_\_\_

Date

\_\_\_\_\_

Study Director

\_\_\_\_\_

Date

\_\_\_\_\_

Auditor, Quality Assurance Unit

\_\_\_\_\_

Date